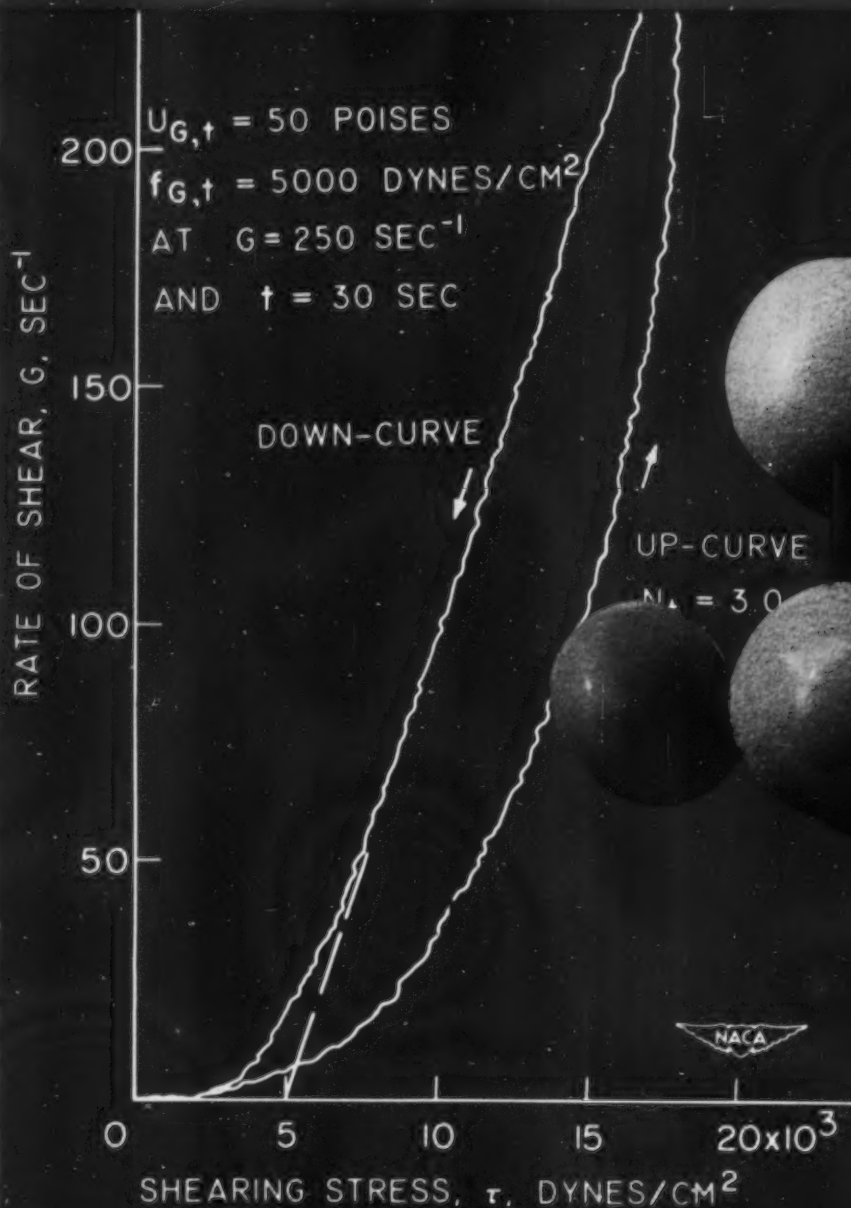


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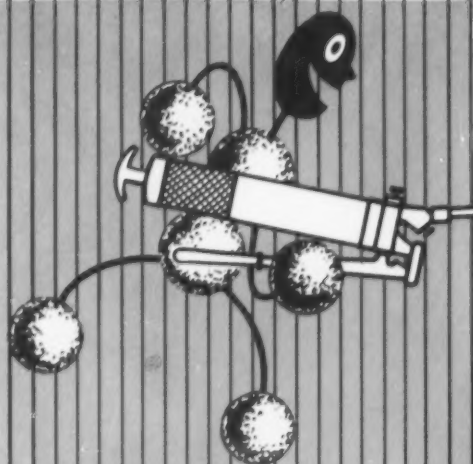
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SYMPOSIUM— Flow Properties of Lubricating Greases . . . Pages 9-40



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President's page

by W. M. MURRAY, President, NLGI

REPORT



President Murray Discusses An Important Subject From the May Board of Directors' Meeting in Colorado Springs



(See page 61)

It rained a good deal of the time during the Board of Directors' meeting at the Broadmoor Hotel in Colorado Springs, which made it very attractive for those of us living in the Southwest. Happily the rain followed us home!

Our meeting was well attended and a considerable volume of work was handled. Among agenda items of particular interest to all of us was that one dealing with a film on the subject of lubricating greases. Vice-President Lane's report for the Motion Picture Committee was comprehensive to the point of finalization.

To produce the film a commitment must be made by the Institute for a definite and sizeable expenditure. The best way to approve the work done so far and to help promote industry progress is for Institute members and friends to place orders for individual prints in advance of completion. Within a short time a letter with descriptive information and an order form will be mailed to members and friends. Further promotion will depend upon your decision. Watch for the mailing and please give the matter your thoughtful consideration. Personally, I am for it!

Many other items could be commented upon but all in all it was a good meeting and I am more and more impressed with the idea that the NLGI is attaining an ever-improving position with industry.



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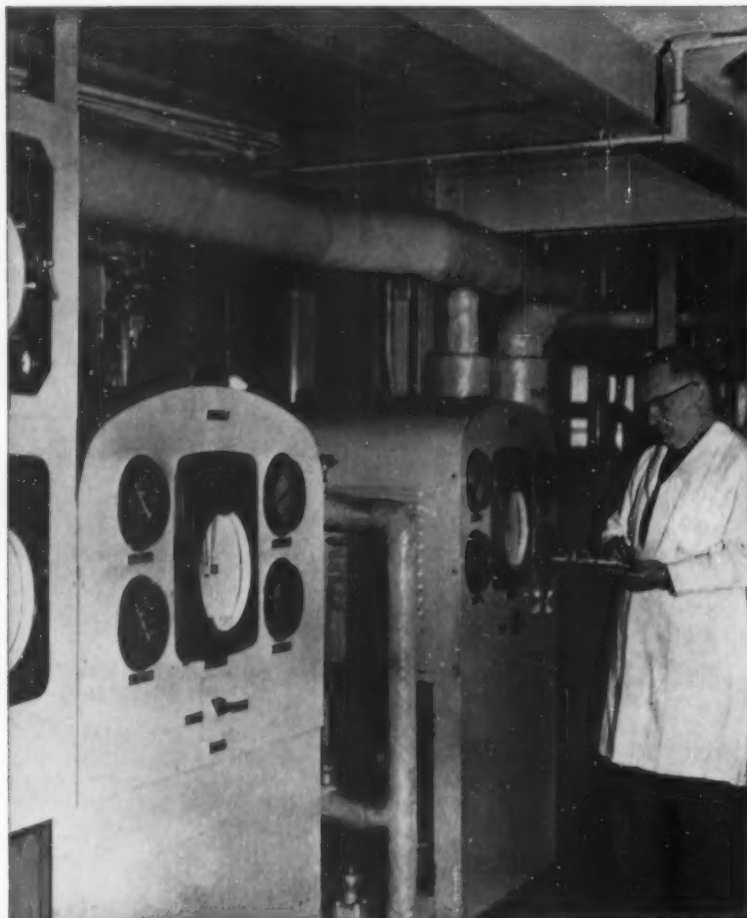


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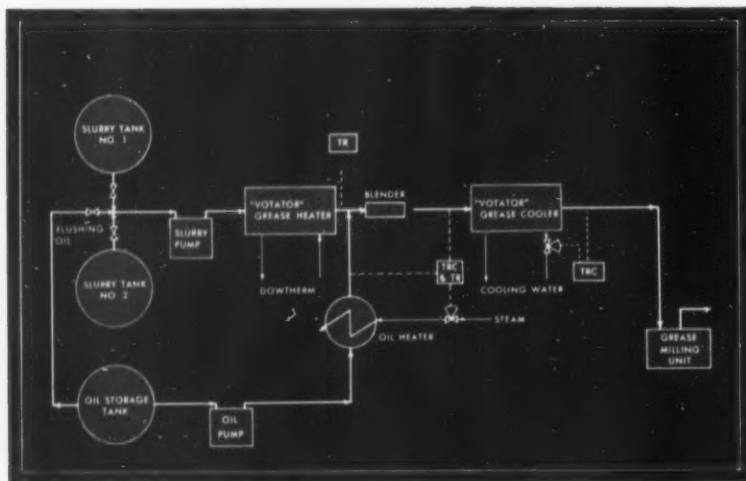
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ABOUT THE COVER

THE PROOF OF SUCCESS of a lubricating grease lies in the characteristics of its flow properties. Determining these characteristics is one of the most difficult problems of our industry. A highly comprehensive symposium on this subject was presented at the NLGI ANNUAL MEETING, October 1955, representing the findings of fifteen authorities in the field. This month's cover is a graph plotting the shear stress of grease, and lithium molecules, a combined illustration representing the first two steps covered in the symposium—basic studies and rheological measurements. The symposium articles appear on pages 9 through 40, with the conclusion, Part III, following next month.



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NLGI SYMPOSIUM

FLOW Properties of Lubricating Greases

Presented at NLGI 23rd Annual Meeting, October, 1955

Introduction By NICHOLAS MARUSOV

The members who have been attending NLGI meetings regularly must realize by now that the flow behavior of lubricating greases differs from that of other lubricants. Furthermore, they must realize that the study of flow properties is a complicated one. Nevertheless, it is because of the peculiar behavior of lubricating greases that there is a Lubricating Grease Industry at all. If lubricating grease flow behavior were similar to oils or other materials, these other materials could and probably would be used instead.

It should also be recognized that the Grease Industry's interest in flow behavior is not merely limited to the passage of material through various conduits as might be used in dispensing equipment or lines leading from grease fittings to the bearings. Flow within these bearings is of prime importance. It is the flow characteristic of a lubricant that determines the degree of successful lubrication that will be attained for any application.

Other grease characteristics, such as bleeding, leeching, and chemical stability, must be considered as secondary. For example, it would make little difference to a customer whether or not a lubricating grease were chemically stable if it would not do a satisfactory lube job. Furthermore, even a grease which is a superior lubricant would be of little value if it could not be forced into a bearing. Slumpability and the ability of a grease to "stay put," as well as its ability to keep metals of bearings separated, can all be defined by constants which describe its flow behavior. Speaking of open antifriction bearings, consider the requirements to which a grease is put. In the region of the rollers or balls, we expect the grease to flow to the area of friction at a very slow rate in order that a continuous supply of lubricant covers mating metal surfaces, yet less than $\frac{1}{4}$ inch away from this region, we expect this same material to "stay put" and act as an effective seal against foreign matter such as water, dust, and other contaminants. On the surface it appears that such specifications would be impossible to achieve and for Newtonian materials, such as oils, it would be impossible, but lubricating greases have for many years been

doing this job satisfactorily. The difficulty in the problem arises from the fact that we are not sure of, or at least cannot express in absolute units, the flow characteristics which define this or other peculiar behaviors of greases. It would also be of tremendous value to know what components in the makeup of greases control the various phases of behavior and to what degree each of these components is effective in doing a satisfactory job.

This Symposium has been designed to include three logical steps which would be developed in any research program. The first step, "Basic Research," merely answers the question in useable absolute units, "Why does this material do what it does?" The second section of the Symposium, called "Rheological Measurements," is an effort to describe in specific mathematical units, "Just what does this particular material do?" The third part of the Symposium, "Practical Application of Rheological Constants," is industry's effort to take the results of these two basic and fundamental studies and apply this information to their specific needs.

By the nature of the problems involved, a large part of this Symposium will involve mathematical and physical studies. The problems are so complex that their solutions require the concentrated thinking of all branches of sciences. It is not the sole intent of this Symposium to be merely a means of reporting progress, although certain findings reported here are of immediate value to many of you. A more important intent of this Symposium is that the information obtained by various investigators can be pooled, discussed, and clarified in order that the next steps of development will be clearly shown. The pooling of information is not restricted to the main speakers. Every company has representatives here who are technically trained and have been working independently with the problems which should be discussed. With a mixed audience as we have here, there are groups primarily interested in marketing. These marketers can find many reasons, as a result of this Symposium, for being confident that their products are the results of the best scientific thinking available.

Left to right, N. Marusov, chairman, H. Eyring, E. O. Forster, L. C. Brunstrum, Ruth N. Weltmann, E. F. Koenig, L. C. Rotter and J. S. Aarons.



I. BASIC STUDIES

Relation of Relaxation Theory to Some Properties of Lubricants

By. H. EYRING, T. REE,
A. FAVA and I. HIGUCHI
University of Utah
Salt Lake City, Utah

ABSTRACT

We have formulated a general reduced equation for transient and steady state behavior of the viscosity of lubricants involving a single relaxation time. For transients it takes the form,

$$-\frac{d \ln \phi}{dt} = 2k_f \sinh b\phi,$$

and for the steady state,

$$\dot{\epsilon} = \frac{\lambda}{\lambda^1} 2k_f \sinh b\phi.$$

This latter equation comes from the former by considering that for the steady state $-d(\ln \phi)/dt$ takes the steady state value $\dot{\epsilon} \lambda^1 / \lambda$. Here ϕ is the ratio of the stress on the system at any time, t , to the initial value, k_f is the equilibrium specific reaction rate and b is a characteristic parameter of the system. The steady state equation is also generalized for systems with multiple relaxation times.

INTRODUCTION

IN THIS PAPER we treat the flow properties of lubricating materials. Thus, the consideration of flow mechanisms in a condensed phase serves appropriately as an introduction.

Many metals flow by movement of dislocation in the crystal lattice.^{1,2} Liquids, in general, can be considered as possessing a "quasi-lattice" structure.^{3,4,5} Thus, we treat the flow of liquids in an analogous fashion to that of solids, the only difference being the presence of more dislocations in liquids than in solids. In fact, if we compare the flow of truly crystalline materials, such as metals, with that of liquids, we find interesting similarities between them. The heats of activation for flow of both states are much lower than might be predicted from the heats of vaporization, and negative entropies of activation are observed for both phases.

We represent the flow process in crystals in Figure 1 schematically. Here, an edge dislocation is shown; ab indicates the slipping plane in a simple cubic lattice, while cd shows a dislocated row of atoms. Thus, one sees readily that the movement of a dislocation by one lattice position requires the movement of only two atoms: one which is moving out of the regular lattice position and requires energy, and the other which is moving into a regular lattice position and give up energy. Because there is co-operative movement of the atoms, the net energy required for the change is much less than that required for flow by evaporation of the individual atoms. The heat of activation for flow, therefore, may be taken as a measure of the degree of cooperation between the atoms taking part in the flow process.

Figure 1 shows also the flow process in liquids provided only that the lattice is now more irregular and there are more dislocations. Thus, flow in liquids is easier than in solids, there being more opportunity for creation or migration of dislocations. The dislocation in liquids is another way of expressing the "hole" in Eyring's theory of liquids,⁶ or the "empty equilibrium sites" in his relaxation theory.^{2b,6,7} In general, the flow processes in solids

and liquids may be visualized as the sudden shifting of some small patch on one side of a shear surface with respect to the neighboring patch on the other side of the surface. Except for the simplest system, this mosaic of patches will be heterogeneous each being characterized by its relaxation time.

GENERALIZED THEORY OF VISCOUS FLOW

Introducing the heterogeneity of flow units (patches), Ree and Eyring⁸ derived the following formula for viscosity, η :

$$\eta = \sum_{i=1}^n \frac{x_i \beta_i}{\alpha_i} \frac{\sinh^{-1} \beta_i \dot{\gamma}}{\beta_i \dot{\gamma}} \quad (1)$$

where,

$$\alpha_i = (\lambda \lambda_2 \lambda_3 / (2kT))_i, \quad (2)$$

$$\beta_i = 1 / \left(\frac{\lambda}{\lambda_1} 2k' \right)_i. \quad (3)$$

The quantity, $\dot{\gamma}$, is the rate of shear, k' is the rate constant for the jumping of the flow unit, λ , λ_1 , λ_2 , λ_3 , are the molecular parameters in the Eyring viscosity theory,^{6,7} and the subscript, i , indicates that the attached quantities belong to the i th group of flow units. The β is dimensionally a time, thus, it is called the relaxation time. X_i indicates the fractional area occupied by the i th unit on the shear surface. Applying the virial theorem, Eyring and Ree⁸ found that

$$\alpha_i = 1 / (29f_0)_i, \quad (4)$$

where, f_0 is the microstress which is frequently found proportional to the initial stress in relaxation experiments, and g is a constant which is approximately equal to the ratio of sound velocity in a gas to that in a solid.

GENERALIZED RATE THEORY OF TRANSIT PHENOMENA

Lubricating materials change their structure in response to changes in such conditions as stress, temperature, pressure, etc. We consider, in general, the rate with which one state of a substance transforms to the other state.

The transformation is due to the driving force impressed by a change of conditions. Let q be the quantity which changes in the transformation. The nonequilibrated system is under a stress. This stress, f , will be represented by:

$$f = c(q - q_e), \quad (5)$$

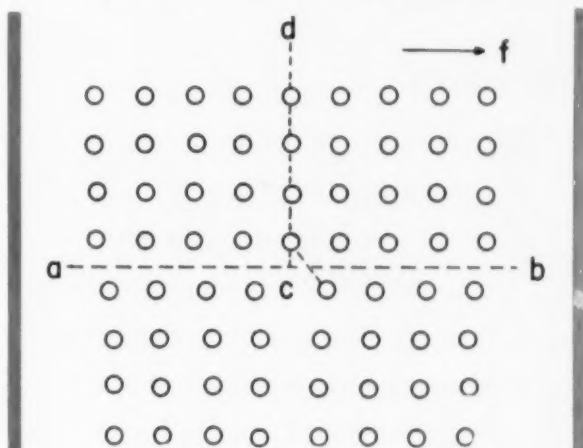
where q and q_e are the quantities at time, t , and at equilibrium, respectively, and c is a proportionality constant. Thus, equation (5) states that the stress is determined by how far the system is from the equilibrium state. The rate with which the quantity, q , decreases is given by:

$$\begin{aligned} -\frac{dq}{dt} &= (q - q_e) k_f e^{\frac{\mu \lambda \lambda_2 \lambda_3 c (q - q_e)}{kT}} \\ &\quad - q_e k_b e^{-\frac{(1-\mu) \lambda \lambda_2 \lambda_3 c (q - q_e)}{kT}}, \end{aligned} \quad (6)$$

where, $q - q_e$ is proportional to the number of units moving in the forward direction toward equilibrium and q_e in the backward direction; k_f and k_b are the specific rate constants of the forward and backward reactions, respectively, and μ is a fractional number arising from the

asymmetry of the barrier. In equation (6) we expect frequently the relationship $\mu = 1/2$ corresponding to a symmetrical barrier. We can now make an important simplification. Since the total free energy driving the system toward equilibrium is $c \lambda \lambda_2 \lambda_3 (q - q_e)$, it follows

Figure 1. Edge dislocation in a simple cubic lattice.



that we must have $(q - q_e) k_f = q_e k_b$ from van 't Hoff's condition that at equilibrium the forward velocity must equal the backward velocity. Incorporating this result yields the very important general equation:

$$\begin{aligned} -\frac{d \ln(q - q_e)}{dt} &= k_f (e^{c\alpha(q - q_e)} - e^{-c\alpha(q - q_e)}) \\ &= 2k_f \sinh c\alpha(q - q_e) \end{aligned} \quad (7)$$

Equation (7) was found for stress relaxation by Ree and Eyring¹⁰ and was successfully applied to the stress relaxation of polyisobutylene, provided that $q - q_e = f$, where f is stress, f_e , the stress at equilibrium, being zero. From equation (4) which followed from the virial theorem, we obtain:

$$\alpha = \frac{1}{2gc(q_0 - q_e)}, \quad (8)$$

where $f_0 = c(q_0 - q_e)$, which follows from equation (5), was introduced. The substitution of equation (8) into (7) yields,

$$-\frac{d \ln(q - q_e)}{dt} = 2k_f \sinh \frac{(q - q_e)}{2g(q_0 - q_e)}. \quad (9)$$

Equation (9) may be written equally well in the reduced form:

$$-\frac{d \ln \phi}{dt} = 2k_f \sinh \phi, \quad (10)$$

where,

$$\phi = (q - q_e) / (q_0 - q_e), \quad (11)$$

ϕ being a dimensionless quantity.

The following is the customary procedure for determining the parameters occurring in equation (10). From the experimental curve of ϕ versus t , the logarithmic rates, $-d \ln \phi / dt$, are obtained. The logarithms of the logarithmic rates thus obtained are plotted against ϕ . If we approximate $\sinh(\phi/2g)$ by $1/2 \exp(\phi/2g)$ in equation

Relation of Relaxation Theory to Some Properties of Lubricants

Continued

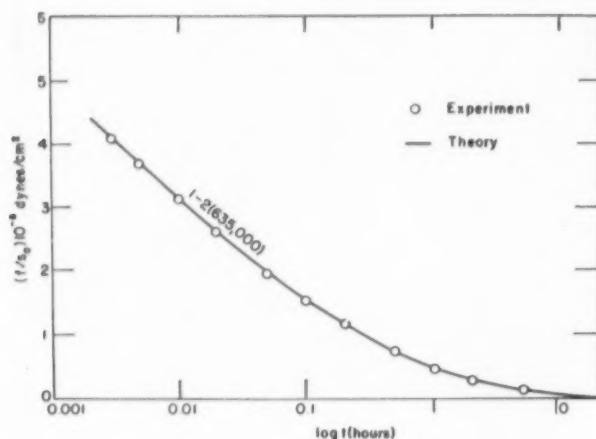


Figure 2. Stress relaxation in polyisobutylene at 30°C.

(10), we obtain the values of $1/2g$ and k_t from the slope of the straight line in the initial period of the plot and from the intercept on the y-axis, respectively. Using the values of $1/2g$ and k_t we now plot $\log [(\ln \phi / dt) + k_t \exp (\phi / 2g)]$ against ϕ to check the parameter, $1/2g$ and k_t , using the logarithmic rates in the whole range of time. The parametric values of 1 and k_t are found the same as before, thus justifying equation (10).

1. Stress Relaxation

Ree and Eyring¹⁰ applied equation (10) for the stress relaxation of polyisobutylene. An example is shown in Figure 2, where the experimental data are due to Andrew, Hofman-Bang, and Tobolsky.¹¹ The theoretical curve is calculated from equation (10), the parametric values are $k_t = 0.407$, $1/2g = 1.38 \times 10^{-6}$. In this case, ϕ is f/s_0 , where f and s_0 are the stress at time, t , and the initial strain, respectively. The quantity, f/s_0 , is related to f/f_0 , the initial stress, f_0 , being related to s_0 by Hooke's law. The stress at equilibrium, f_e , is zero in this case. One sees that

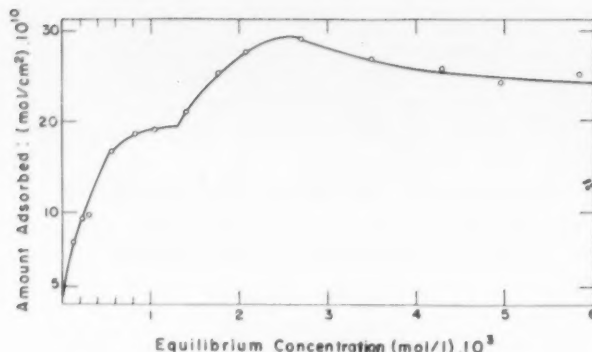


Figure 3. Adsorption isotherm of DBSNa on nickel at 29.5°C.

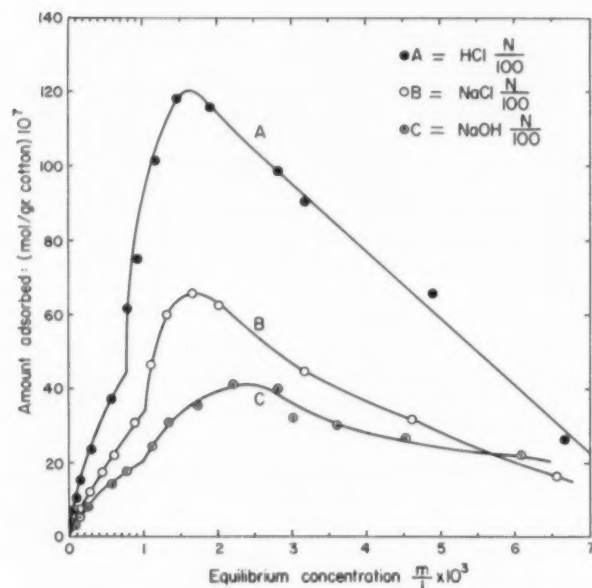


Figure 4. Adsorption isotherms of DBSNa on cotton at 30°C.

the agreement between experiment and theory is satisfactory.

2. Rates of Adsorption and Desorption

Successful boundary lubrication depends on adsorption of the lubricant at the interface. It is thus desirable to understand the equilibrium and kinetic behavior of lubricant molecules in a surface. In this laboratory studies¹² have been made of the adsorption of the detergent, sodium dodecyl benzene sulfonate, from water on nickel and on cotton cloth. The detergent was made radioactive by using S^{35} . In Figure 3 one sees the equilibrium adsorption of detergent on nickel metal. There is a characteristic break occurring at a detergent concentration of about 10^{-3} mole/l. A similar break has been observed by Daniel¹³ for the adsorption of octadecyl alcohol on nickel powder. Daniel interpreted this behavior as the lying down of the alcohol molecules on the surface at low concentrations. He estimated each such molecule occupied about 100 \AA^2 . At the higher concentration the upended molecules were estimated to occupy about 20 \AA^2 .

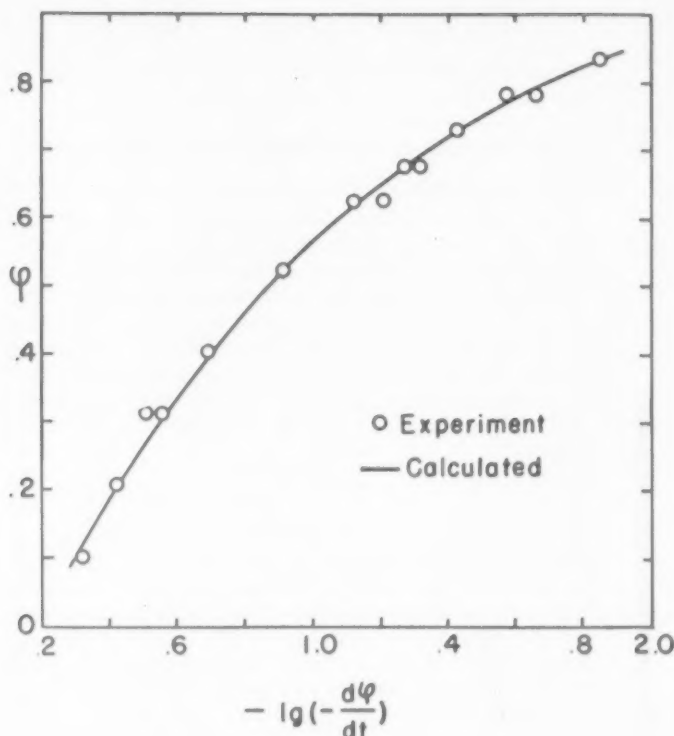


Figure 5. Adsorption rate of DBSNa on cotton at 3°C.

The detailed study¹¹ of sodium dodecyl benzene sulfonate on cotton cloth shows the same kind of break observed with nickel. Fava and Eyring repeated the same experiment in this laboratory and found the same kind of breaks as exemplified in Figure 4.

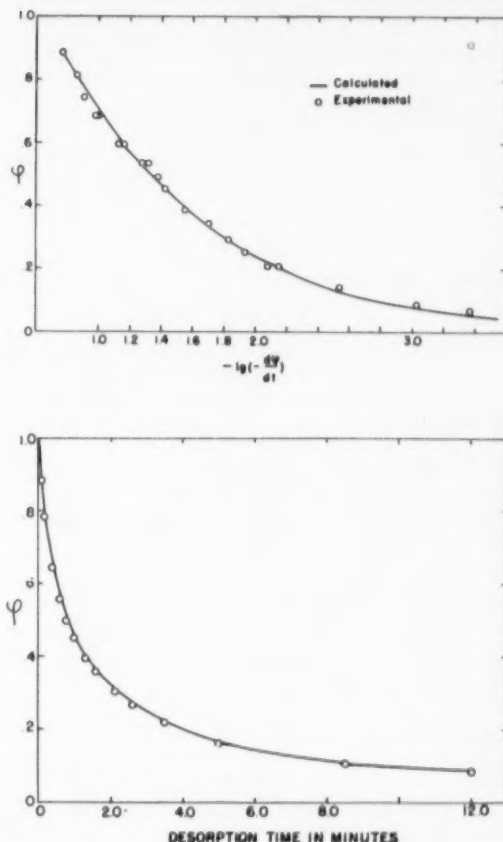
In addition, Fava and Eyring studied the rate of adsorption on cotton cloth of sodium dodecyl benzene sulfonate from solutions varying in concentration from 5.6×10^{-5} to 3×10^{-3} molal (c.f. Fig. 5). The exciting result found was that all of these observed rates fit a single reduced equation within experimental error. The equation obeyed is:

$$-\frac{d\ln(1-\phi)}{dt} = 2k' \sinh \frac{1-\phi}{2g} \quad (12)$$

Equation (12) is readily obtained from equation (10) by replacing ϕ in the latter by $(1-\phi)$. The simple replacement is justified because in the adsorption the stresses, f and f_0 , on the system are $c(q_e - q)$, and $c(q_e - q_0)$ at time, t and zero, respectively, from equation (5). In equation (12) ϕ is the ratio of the amount, q , adsorbed

Figure 6. Desorption of DBSNa from cotton at 2°C.

Figure 7. Bottom, desorption rate of DBSNa from cotton at 29.5°C.



at time, t , to the final equilibrium amount, q_e , at infinite time: k' is, of course, the specific rate of going on to the surface when ϕ is unity. The dimensionless quantity $1/2g$ is found independent of both temperature and concentration.

More amazing still is that desorption of the sulfonate into pure water follows precisely the completely analogous reduced equation (10), i.e.,

$$-\frac{d\ln\phi}{dt} = 2k'' \sinh \frac{\phi}{2g} \quad (13)$$

All the symbols of equations (12) and (13) have the same meaning and values except that in (12) the specific rate constant, k' , is for adsorption while in (13), the rate constant k'' is for desorption. Two example of the desorption of sodium dodecyl benzene sulphonate from cotton are shown in Figures 6 and 7. The numerical values are $1/2g = 1.26$; $k'_{30^\circ} = 1.17 \times 10^{-3} \text{ sec}^{-1}$; $k''_{2^\circ} = 7.37 \times 10^{-4} \text{ sec}^{-1}$; $k'_{30^\circ} = 4.65 \times 10^{-3} \text{ sec}^{-1}$; $k'_{3^\circ} = 3.35 \times 10^{-3} \text{ sec}^{-1}$. Or at all temperatures $k' = \frac{kT}{h} \exp$

$$(-62.3/R) \exp \left(\frac{-2,075}{RT} \right) \text{ and } k'' = \frac{kT}{h} \exp \left(\frac{-62.0}{R} \right) \exp \left(\frac{-2,720}{RT} \right)$$

From this we obtain for the heat of adsorption $\Delta H = 2,075 - 2,720 = -645$ calories. From equilibrium data the value $\Delta H = -773$ calories is obtained.

The desorption of alkali metals from tungsten surfaces was studied by Higuchi, Ree and Eyring,¹⁵ and they found that the abbreviated form of equation (10),

$$-\frac{d \ln \phi}{dt} = k_f e^{b\phi} \quad (14)$$

where b is a constant, and,

$$\phi = q/q_0 = \theta/\theta_m$$

holds exactly in this case. Equation (14) is also applicable for the desorption of barium and strontium from tung-

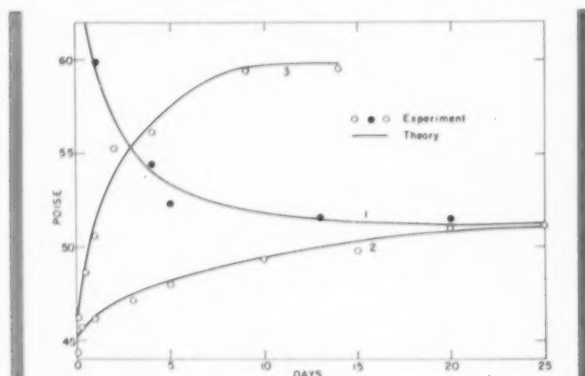


Figure 8. Change of viscosity with time. Curve 1: Pennsylvania motor oil V-4 chilled to -80°C , then kept at 0°C . Curve 2: Same oil cooled from room temperature to 0°C , then kept at 0°C . Curve 3: Persian lubricating oil kept at -10°C .

sten surfaces.¹⁶ Thus, we see that equation (10) describes very generally the relaxation of a condensed system toward equilibrium.

Transient Phenomena in Lubricants

The effect of thermal history on the low-temperature viscosity of partially crystallized lubricating oils has been studied quite thoroughly by Jordachescu.^{17,18}

The experimental data in Figure 8 are taken from Jordachescu's paper. One sees that several days may elapse before the viscosity reaches a constant value, i.e. before crystallization and structure formation are completed. The fact that approximately the same viscosity value was finally reached at 0°C , regardless of whether the oil has previously been heated to $+60^\circ\text{C}$ or cooled to -80°C , shows that a true equilibrium state exists which can be approached from both directions.

We calculate the rates of change of viscosity from equation (10). The theoretical curves are calculated from the following formula which is an abbreviated form of (10):

$$-\frac{d \ln(n-n_e)}{dt} = k_f e^{b(n-n_e)} \quad (15)$$

The parametric values are as follows:

	k_f	b
Curve 1	2.61	0.776
Curve 2	0.391	2.88
Curve 3	2.41	0.456

Here again, the change of viscosity is a relaxation toward equilibrium arising from a driving force suddenly imposed through a change in the condition imposed on the oil.

STEADY STATE PHENOMENA FOR LUBRICANTS

1. Pressure Effect on the Viscosity of Lubricating Oils

The effect of pressure on viscosity of lubricating oils has significant importance in practice. Consequently, many elaborate investigations have been carried out in this field.¹⁹ One example is shown in Figure 9, where the rates of shear, $\dot{\gamma}$, of Sperm oil at 0°C under various pressures ranging from 9,500 to 29,000 psi. are plotted against the shear stress, f . The experimental data in Figure 9 are taken from the paper of Norton, Knott and Muen-ger.²⁰ The theoretical curves are calculated from the equation:

$$f = \frac{1}{\alpha} \sinh^{-1} \beta \dot{\gamma} \quad (16)$$

Equation (16) is the simplest case of equation (1), i.e. there is only one kind of flow unit. The parametric values are tabulated in Table I. One sees that at low pressures sperm oil behaves almost like a Newtonian body while it behaves as a marked non-Newtonian body at high pressures. From Table I, one sees that both factors, $1/\alpha$ and β , change with pressure, the change of relaxation time, β , being most striking.

The effect of pressure on viscosity has been studied by Ewell and Eyring,²¹ and Frish, Eyring, and Kincaid.²² The pressure effect has been explained by the increased

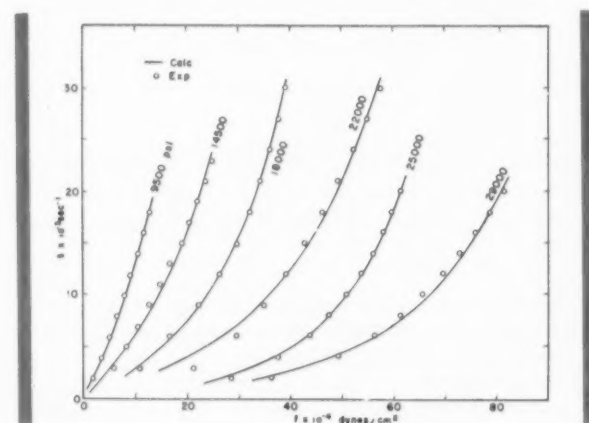


Figure 9. Pressure effect on the viscosity of sperm oil at 0°C .

volume of the activated complex for flow, consequently, by the increase of the free energy of activation with pressure, i.e.

$$\Delta F^\ddagger = \Delta F^\ddagger_1 + p \Delta \bar{V}^\ddagger \quad (17)$$

where, $\Delta \bar{V}^\ddagger$ indicates the average increased volume of the activated complex. From the β values in Table I, we obtain the value of $\Delta \bar{V}^\ddagger$ as 50 cc/mole. Sperm oil is

complex in nature; so that neither the composition nor molecular volume, V , is known. According to Eyring, Ewell, Frish and Kincaid,^{21,22} the ΔV^\ddagger is equated to V/n , n being a numerical number; it is about 7 for non-associated liquids and about 22 for metals. Lacking an exact value for V , n can not be calculated here.

2. Effect of Composition on the Viscosity of Greases

Blott and Samuel²³ measured the flow rates of the lime base greases which are a kind of colloidal solution of calcium soap in oil. Their results are well expressed by

$$f = \frac{x_1 \beta_1 \dot{\gamma}}{\alpha_1} + \frac{x_2}{\alpha_2} \sinh^{-1} \beta_2 \dot{\gamma}, \quad (18)$$

which is an abbreviated form of equation (1) first used in this connection by Powell and Eyring.²⁴ The parametric values are listed in Table II. The theoretical curves calculated from (18) using the parametric values in Table II are compared with the experiment in Figure 10. They used four different kinds of grease, A, B, C, and D; the composition of the greases is shown in Table II.

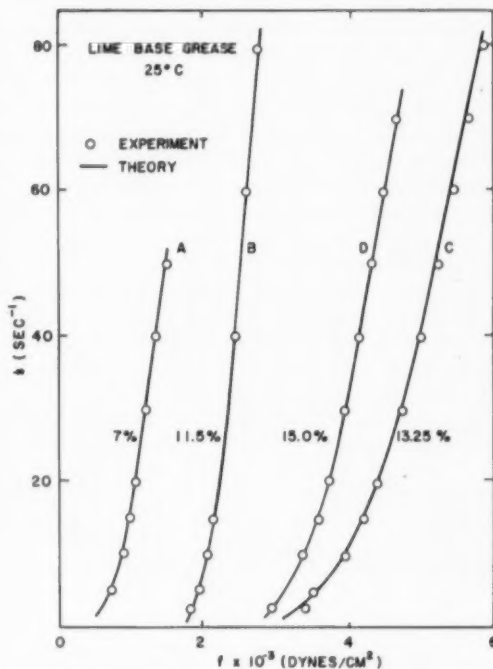


Figure 10. Shear rate vs. shear stress for lime base grease. The percentages of lime in grease, A, B, C, and D are 7, 11.5, 13.25, and 15.0, respectively.

The viscosities of the four greases are in the order $C > D > B > A$, in the range of $\dot{\gamma}$ covered by these experiments. Among the factors in Table II, only the values of x_2/α_2 accord with this order, while the factor β bears no relation to this order. We reached the same conclusion earlier with respect to the effect of solvents on the viscosities of solutions of styrene and polyisobutylene.⁸

With the development of this general theory of transient and steady state behavior of lubricants in terms of molecular concepts one can hope to more rationally relate molecular structure and lubricant performance.

Acknowledgement

We wish to thank the Office of Naval Research, the California Research Corporation and the Foreign Operation Administration of the U. S. Government for support during this investigation.

TABLE I
Effect Pressure on the Viscosity of Sperm Oil at 0°C

Pressure (P.s.i.)	Pressure (atm)	$\frac{1}{\alpha}$	β
9500	655.0	$1.02 \cdot 10^5$	$0.091 \cdot 10^{-3}$
14500	999.7	$1.35 \cdot 10^5$	$0.130 \cdot 10^{-3}$
18000	1241.0	$1.45 \cdot 10^5$	$0.250 \cdot 10^{-3}$
22000	1516.8	$1.97 \cdot 10^5$	$0.300 \cdot 10^{-3}$
25000	1723.7	$1.554 \cdot 10^5$	$1.350 \cdot 10^{-3}$
29000	1999.5	$2.04 \cdot 10^5$	$1.320 \cdot 10^{-3}$

TABLE II
The Parameters for the Lime-Base Greases at 25°C

Kind of Grease	Per-centage of Lime	η oil at 25° Poises	$\frac{x_1 \beta_1}{\alpha_1}$ at 25° Poises	$\frac{x_2}{\alpha_2}$ dynes/cm ²	$\beta \times 10^{-3}$ sec
A	7	2.74	9.94	115	0.0456
B	11.5	0.29	4.80	140	86.6
C	13.25	1.52	12.6	475	0.152
D	15.0	0.29	11.34	297	2.68

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I. INTRODUCTION

The structure of greases has been the concern of many investigations and formed the subject of a symposium at the 1954 annual meeting of the National Lubricating Grease Institute¹. The general consensus of the discussion at that symposium was that the structure of a grease can best be visualized as a three dimensional network of soap fibers with the lubricating oil trapped between fibers. The ease with which such a structure will flow under an applied force will depend on the rigidity of the soap fiber network. This rigidity of the soap structure will be determined by the forces holding the soap fibers together. To predict a grease's tendency to yield to stress, and thus to flow, it is necessary to know what the forces are that hold the fibers together.

II. SUMMARY

The structure of the unit cell in metal stearates has been postulated, and confirmed experimentally by X-ray diffraction techniques. The unit cell is the same for a given soap whether in the form of crystals or in the form of soap fibers. The soap molecules are held together by ionic and Van der Waal forces. These forces vary directionally. Between adjacent molecules in the same row, the forces are 10 to 100 times stronger than the forces perpendicular to the row. The dielectric strength and viscosity of a medium surrounding soap molecules weakens these attractive forces, with the result that crystal growth will occur chiefly in the direction of the strongest force, resulting in fiber formation.

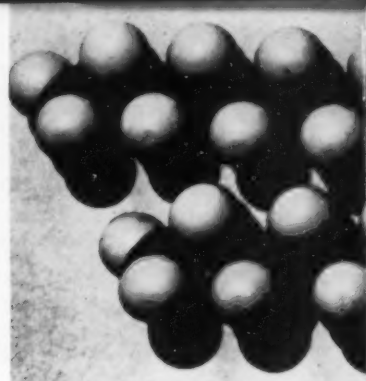
Adjacent soap fibers in a grease are held together by the same type of forces found within the individual fibers. The process of network formation is similar to the mechanism of polymerization of tri-functional molecules. The forces vary directionally, and the weaker linkages of the network may be therefore disrupted by low rates of shear. High rates of shear supply sufficient energy to break the soap fibers, as well as pull the fibers apart from each other. More quantitative calculations will have to be made to predict accurately the flow properties of greases from a knowledge of the structure of their soap systems.

III. DISCUSSION AND RESULTS

Most metal salts of fatty acids commonly used in the manufacture of greases are crystalline in nature. Upon their dispersion in a hydrocarbon medium, they form fibers which stick to each other and thus form a network. To understand how a soap can produce fibers and thus thicken an oil, it is necessary to compare the arrangement of the soap molecules in the fiber with that in the original soap. This can be done by comparing X-ray diffraction patterns and electron micrographs of the original soap crystals with those of the soap fibers present in a grease.

A. The Unit Cell

The basic structural unit of a crystalline material, the unit cell, can usually be determined from the position of the X-ray diffraction lines using the Debye-Scherrer powder technique². All the metal soaps investigated produce over 40 diffraction lines and belong to crystal systems of low symmetry. To index such a large number of lines and hence to deduce the dimensions of the unit cell requires special graphic methods such as those developed

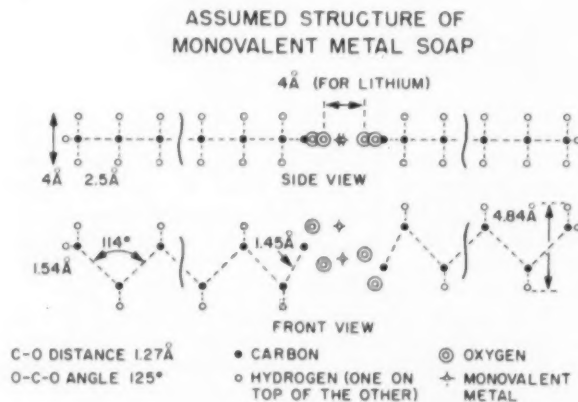


FIBERS, FORCES

by Vand³. These methods take advantage of the fact that in soaps one edge of the unit cell is much longer than the other two and corresponds to the length of a double molecule of soap.

The internal structure of the unit cell, i.e. the arrangement of the hydrocarbon chain, the carboxyl group, and the metal within the unit cell, is determined from the intensity of the diffraction lines. Usually such structure determinations are made with single crystals of the material under investigation using complex mathematical computation techniques such as the Fourier synthesis⁴. In the case of the metal soap studied, it is very difficult to grow such single crystals and hence very few attempts have been made in the past to determine their structure. By taking advantage of the aforementioned fact that one edge of the unit cell is much longer than the other two, it is possible to obtain considerable structural information from powder camera diffraction pictures. Instead of the customary three dimensional Fourier synthesis, only a one dimensional synthesis is made along the long edge of the unit cell. Using the knowledge of the chemical composition of the soap molecules and the probable arrangement of the constituent atoms within the molecule a structure is assumed as shown in Figure 1. In the one

Figure 1



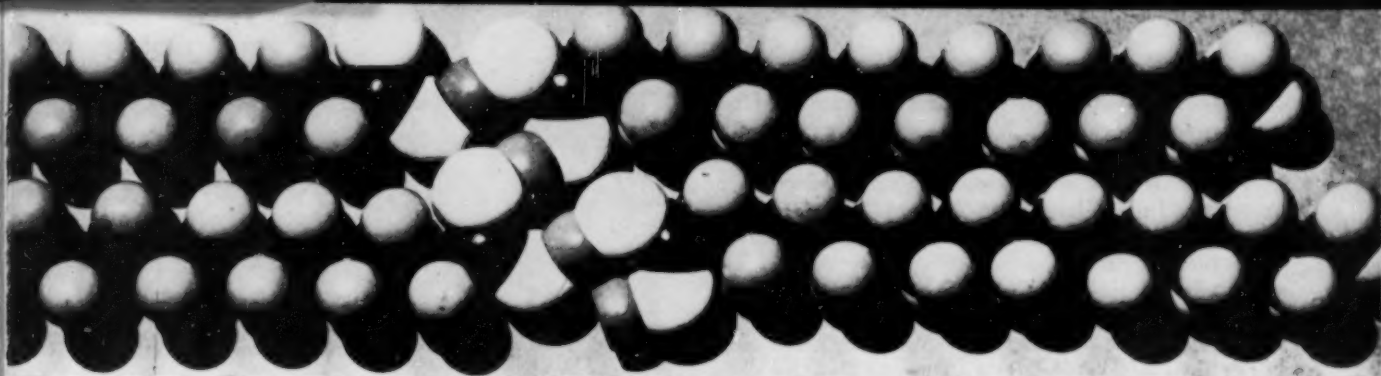


Figure 3. Most stable configuration of lithium stearate molecules within a parallel row.

and FLOW

By E. O. FORSTER—J. J. KOLFENBACH—H. L. LELAND
Esso Research and Engineering Company

dimensional Fourier synthesis all the atoms are projected onto the long edge of the unit cell and the intensities of various X-ray reflections related to this long edge are calculated. The degree of agreement between the calculated and observed intensities is then a measure of how closely the assumed structure corresponds to that actually existing in the crystal. The intensities of the first five reflections were calculated for various metal stearates and found to agree well with those actually observed as shown in Table 1. This good agreement indicates that the assumed crystal structures correspond closely to the actual structure. Thus, information is obtained on the relative positions of the metal atom, the carboxyl group, and the hydrocarbon chain. The results of this calculation do not, however, define uniquely the arrangement of the hydrocarbon chains with respect to each other. In general, these hydrocarbon chains can be arranged in several different ways as shown in Figure 2. From considerations of the dimensions of the unit cell only the first two arrangements (a) and (b) appear to be present⁵. In one case these chains belonging to adjacent molecules in the same row have their carbon planes in parallel with each other as shown in (a) of Figure 2. In the other case the chains

belonging to adjacent molecules in the same row are in alignment with each other as shown in (b) of Figure 2.

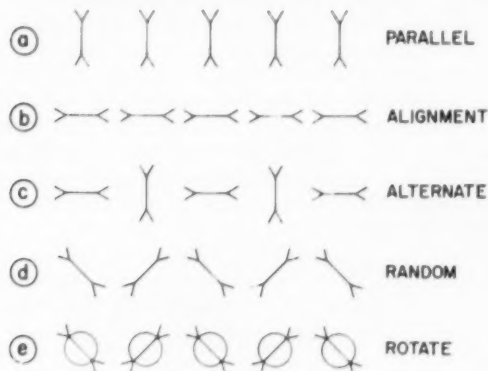
B. Forces in Soap Crystals

From a knowledge of this arrangement of the soap molecules in the unit cell, the nature and magnitude of forces holding these molecules together are determined. Two types of forces hold molecules together to form crystals. One is essentially ionic in nature while the other is due to Van der Waal forces. In the case of the soap molecules, the ionic forces are derived from the attraction of the negatively charged carboxyl group and the positively charged metal ion and are inversely proportional to the distance between these charged particles. The Van der Waal forces are due to induced dipole-induced dipole interaction between neighboring methyl and methylene groups and are inversely proportional to the sixth power of the distance between the interacting groups.

In the unit cell the carboxyl groups and the metal ions are arranged in such a manner that the distances between oxygen and metal ions belonging to adjacent molecules in the same row are shorter than those between oxygen and metal ions belonging to molecules in neighboring rows. Consequently, there will exist two ionic forces, a stronger one operating within the same row between adjacent molecules and the other weaker one operating in a direction perpendicular to the first between neighboring rows. The magnitude of the Van der Waal forces will depend on the relative packing of the chains. These forces will be strongest if the planes containing the zig-zag chains of the carbon atoms are aligned ("b" in Figure 2) and weakest if these planes are parallel to each other ("a" in Figure 2). These forces will tend to bring adjacent hydrocarbon chains within the same row as close as possible to each other. This would, however, bring adjacent oxygen atoms so closely together that their repulsion would be greater than the attraction between the metal ion and the oxygens. In the case of monovalent metal soap such as lithium stearate, this is avoided by placing one oxygen of the carboxyl group above and the other below the plane formed by the carbon atoms of the hydrocarbon

Figure 2

PACKING OF CH_2 CHAINS IN A PARALLEL ROW



chain as shown in Figure 3. The displacement of the pulsive force without reducing the distance between oxygen atoms out of plane increases the distance between oxygens of adjacent molecules and thus reduces their re-oxygens and metal ions. In this arrangement, the molecules lying in the same row have their hydrocarbon chains tilted to the region containing the metal ions. The angle between the hydrocarbon chains and this region varies from metal soap to metal soap. Within the ionic region the metal ions lose their identity and are equally shared by adjacent fatty acid radicals. Thus the total attractive forces operating in a soap crystal will be stronger within a row of molecules than between rows of molecules. If rows of molecules contact each other only through the terminal methyl groups of their hydrocarbon chains, the only attractive force will be of the Van der Waal type, and its magnitude will depend on the number of methyl groups interacting with each other. The crystal structure of a soap appears to resemble closely that of such layer crystals as talc, mica or graphite in which molecules within a layer are held by strong forces while only weak forces operate between layers. This arrangement accounts for the well known tendency of soap crystals to split very easily along planes parallel to rows, a property so prominent that the splitting takes place when the crystals are rubbed between the fingers, giving rise to a "fatty" touch.

C. Fiber Formation

Once the arrangement of the soap molecules in the unit cell and the forces operating among them had been deduced, it became possible to visualize the process of fiber formation by electrostatic forces between oppositely charged ions and by Van der Waal forces. Of these forces, the electrostatic ones far outweigh the Van der Waal forces. The magnitude of the attractive forces will depend on the individual soap species and will be different in the various directions. In the case of a monovalent metal soap such as lithium stearate the total attractive force between adjacent soap molecules lying in the same row was calculated to be about 10 times as strong as those operating between soap molecules lying in successive rows and about 100 times as strong as that operating between pairs of terminal methyl groups. These calculations are based on the treatment of ionic and dipole interactions as reported in the paper by Moelwyn-Hughes et al⁶. The results are shown in the following table.

Total Attractive Forces Between Soap Molecules	
Within the same row	30 Kcal/mol
Between neighboring rows	3 Kcal/mol
Between terminal methyl groups	0.3 Kcal/mol

1. Theoretical Considerations

The effectiveness of the electrostatic forces depends on the dielectric properties and the viscosity of the medium surrounding the charged ions. Hydrocarbons have a low dielectric constant and will not isolate ions as would a medium of high dielectric constant such as water. Also,

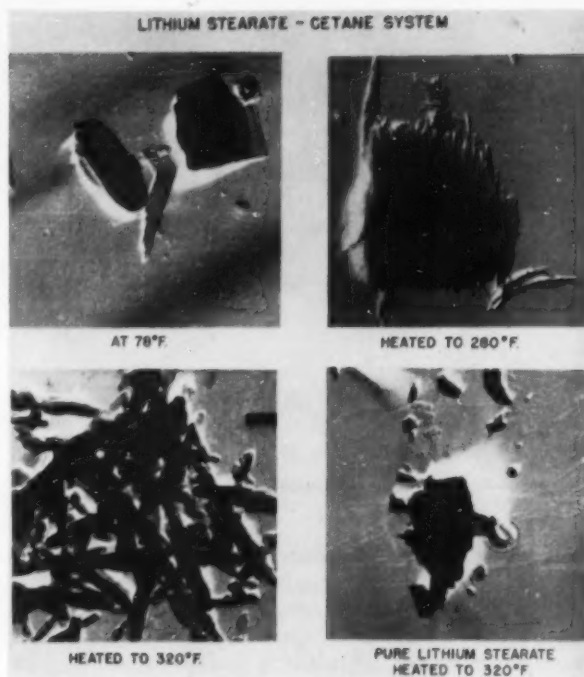
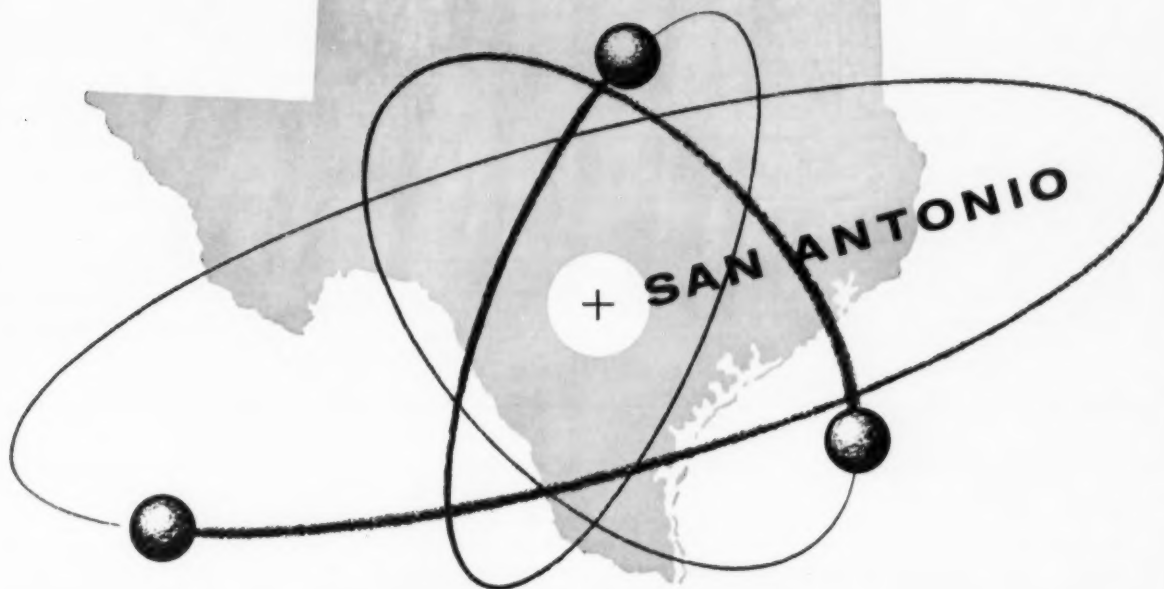


Figure 4

the hydrocarbon medium has a high viscosity and interacts with the hydrocarbon chain of the fatty acid radical. As a consequence of the low dielectric constant of the medium, soap molecules will interact most readily with each other to form dimers in which the carboxyl groups and the metal ions will be facing each other. In order to achieve a crystalline arrangement, the soap dimers will have to combine with each other and to do this the ionic forces of the dimers will have to squeeze away hydrocarbon molecules of the medium which have the tendency to interact with the hydrocarbon tails of the dimers. The frequency of recombination of soap dimers to form a crystalline material will depend on the concentration of free soap dimers, the temperature of the medium, and the magnitude of the attractive forces. The concentration of the free soap dimers can be increased by heating a mixture of soap and hydrocarbon medium and dissolving more and more soap dimers or by breaking soap crystals into small units by the use of high rates of shear. Heating will also lower the viscosity of the medium and reduce the tendency toward interaction between soap and medium. At the same time heating, however, will reduce the tendency of the soap dimers to form a crystalline arrangement since their kinetic energy will increase and eventually become greater than the attractive forces. Most recombinations will occur in the direction of the strongest force because it will be the only one large enough to displace the interfering hydrocarbon molecules. Few recombinations will take place in the direction of the second, weaker force and very few will occur in the direction of the weak Van der Waal forces. The recrystallization will, therefore, result in long, thin thread-like crystals rather than the original platelet. Agglomerations of these thread-like crystals are commonly referred to as soap fibers.

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PERIODIC CLASSIFICATION OF THE ELEMENTS																		
GROUP	I _a	II _a	III _a	IV _a	V _a	VI _a	VII _a	VIII _a	I _b	II _b	III _b	IV _b	V _b	VI _b	VII _b	VIII _b		
PERIODS	1	H																He
	2	Li	Be														C	Ne
	3	Na	Mg														Al	Ar
	4	K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Kr
	5	Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	Xe
	6	Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	Rn
	7	Fr	Ra	Ac	Rare Earths			Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Lu
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Lithium differs in metallurgy...

inasmuch as the affinity of Lithium for oxygen, for example, is being utilized to reduce porosity in copper and copper alloy castings. Recent research has revealed that Lithium will produce brazing alloys with self-fluxing properties and increase the wetting ability of these alloys.

Lithium differs in inorganic chemistry . . .

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Lithium differs in heat transfer . . .

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2. Experimental Confirmation

Confirmation of this fiber forming mechanism is found from studies of soap dispersions in hydrocarbon media using the electron microscope and X-ray diffraction. In Figure 4 are shown electron micrographs of a dispersion of lithium stearate in cetane before and after heating to various temperatures. As the temperature increases above a critical point, the soap dissolves nearly completely in the cetane and upon cooling recrystallizes in the form of fibers. In the absence of a hydrocarbon medium, no fibers are formed and the general platelet-like shape is preserved.

The X-ray diffraction photographs reveal that the unit cell structure of the soap is slightly but irreversibly changed upon heating and these changes occur whether the soap is heated by itself or in the presence of a hydrocarbon medium. These numerical results are shown in the following table where the long "d" spacings of several soaps are listed before and after being heated in the presence and absence of a hydrocarbon medium.

The "d" Spacings of Various Soap Dispersions

	"d" Spacing (in Angstrom Units)	
	Before Heating	After Heating
Lithium Stearate pure	41.5	42.0
25% Lithium Stearate in Cetane	41.5	42.0
12% Lithium Stearate in Oil*	41.5	42.0
Sodium Stearate pure	45.1	43.0
25% Sodium Stearate in Cetane	45.1	43.1
12% Sodium Stearate in Oil*	45.1	43.0
Calcium Stearate pure	48.8	49.6
25% Calcium Stearate in Cetane	48.8	49.6
12% Calcium Stearate in Oil*	48.8	49.6

*A naphthenic distillate of 55 S.S.U./210°F. viscosity.

The selection of this "d" spacing as a measure of internal structural changes is based on the fact that it is more sensitive to them than the other dimensions of the unit cell. This is illustrated in Figure 5 where X-ray diffraction photographs are shown for samples of sodium stearate heated to various temperatures. The main structural changes that occur are of the order of a few per cent at best and amount to changes of a few hundredths of an Angstrom unit for the edges a and b of the unit cell but are of the order of one or several Angstrom units for the long "d" spacing. These internal changes are caused by a rearrangement of the soap molecules in the unit cell

Figure 5

X-RAY DIFFRACTION OF SODIUM STEARATE-CETANE SYSTEM USING FeK α RADIATION

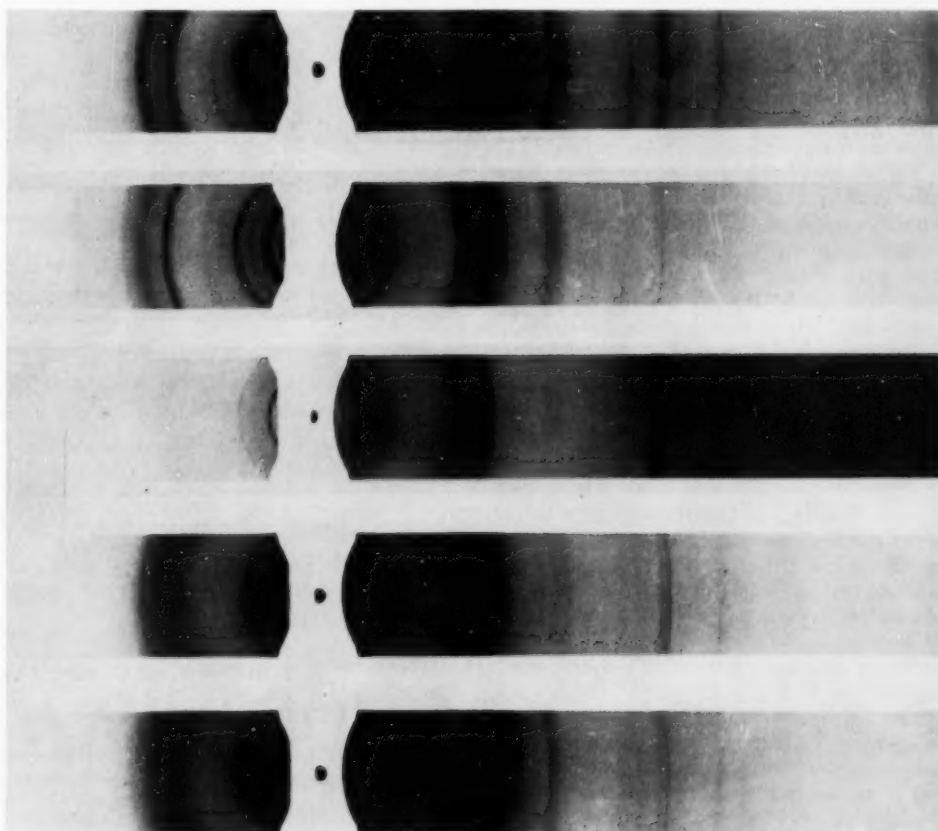
1. PURE SOAP

2. 25% SOAP IN CETANE BLENDED COLD

3. 25% SOAP IN CETANE HEATED TO 120°C

4. 25% SOAP IN CETANE HEATED TO 180°C

5. PURE SOAP HEATED TO 180°C



as a result of heating. With an increase in temperature the volume occupied by the hydrocarbon chains increases and the packing of the soap molecules will change so as to achieve the most stable structure possible under these conditions. Upon cooling the molecules lose their kinetic energy and are no longer able to regain their original position. But they will assume the same unit cell structure, whether this process takes place in the absence or presence of a hydrocarbon medium.

D. Grease Formation

In the course of fiber formation fibers will get in contact with each other. At the points of contact attractive ionic and Van der Waal forces will become operative and hold these fibers together. The extent to which these forces will be effective will depend on the way the fibers contact each other. The three basic types of fiber contact are illustrated in Figure 6. In the first case only the hydrocarbon tails will be in contact with each other and the effective force will be essentially of the Van der Waal type. In the second case, hydrocarbon chains and ionic groups interact with hydrocarbon tails and the effective force will be the result of both Van der Waal and ionic forces with the latter contributing only little. In the last case both ionic groups and hydrocarbon chains interact and the resulting force will be the result of Van der Waal and ionic forces with the latter predominating. The magnitude of these attractive forces will depend in each case on the angle under which the two fibers cross and on the width of the fibers in the first two cases. Assuming the angle and width to be the same in each case, the attractive forces will be greatest in the first case and about the same in the other two. If the angle between contacting fibers becomes zero, it will represent a piling of one fiber on top of another in the first case, creating a new fiber with double the height of the first. In the second case, there will be no change, and in the third case, there will be an increase of the fiber width to twice the original.

The forces holding adjacent fibers together are the same as those holding the soap molecules within the fiber. The formation of the three dimensional network can be looked upon as the result of a polymerization process. The soap dimers are considered to be trifunctional monomers with the previously described three forces which hold fibers together corresponding to three different functional groups. The polymer growth will take place along the various functional groups depending on their relative reactivity and hence, most of it will occur in the direction of the strongest force and less in the other two directions, as would be predicted from physical chemistry.

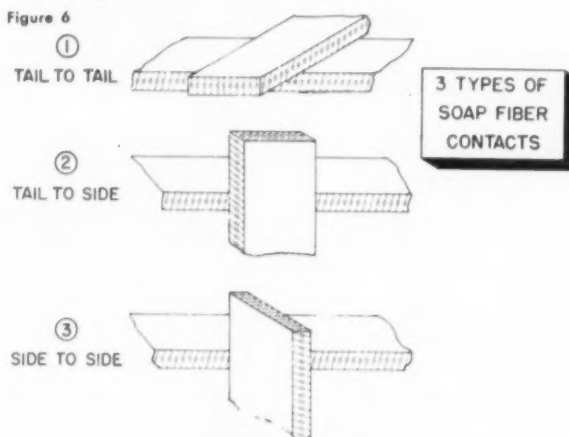
E. Effect of Shear on Grease Structure

On the basis of this information the effect of shearing on the grease structure can be readily understood. Depending on the rate of shearing the structural breakdown can range from a mere breaking of contact points between fibers at low rates, to the decrease of fiber height, width, or length at increasingly higher rates¹. The various stages might be best illustrated by the following examples: When a grease is forced through a relatively large pipe or opening it moves with what is commonly referred to as "plug flow." Breakdown occurs only in the close vicinity of the walls and is essentially restricted to the breaking of fiber contacts in that area while the bulk of

the grease moves as a whole unchanged. If the diameter of the pipe becomes very small, the "wall effect" will become more important and will be accompanied by a decrease in fiber height. This means that layers of soap dimers will slide over each other in order to facilitate movement. The fibers will also arrange to have their long axes parallel to the direction of flow. Eventually at very small openings, at very high rates of shear, the fibers will also be reduced in width and length in order to lower their resistance to movement. A shortening of the fibers might also be expected to occur under conditions of turbulent flow.

The ability of a grease to regain its consistency after extensive breakdown is referred to as thixotropy, and will depend on the soap type as well as the long range forces² that will bring fibers close enough to each other to reform contact points. Once the fibers are brought into close contact with each other by either long range forces, mixing, or heating, new contact points will form. Since the number of soap fibers could be increased by the breakdown of the original fibers more contact points could be formed and the grease could appear harder than before breakdown occurred. This concept is in good agreement with the observed softening of greases under low rates of shear and hardening under high rates of shear³. Thus the structural information gathered so far seems to account satisfactorily for the formation of fibers in a grease and explains why these fibers stick together to form a three dimensional network. It is also consistent with the known behavior of greases under shear. More quantitative calculations will have to be made before it will become possible to predict accurately the flow properties of greases.

Figure 6



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$$f = Prc/2L$$

$$m = Q/\pi r^3 f \alpha$$

$$\eta_p = 1/m = (P/Q) \frac{\pi r^4 c \alpha}{2 L}$$

II Rheological Measurements

Capillary Viscometry of Lubricating Grease

By L. C. BRUNSTRUM and R. H. LEET
Standard Oil Company, Whiting, Indiana

INTRODUCTION

ONE OF THE DEFINING characteristics of lubricating grease is the ability to flow. Under most conditions, the nature of this flow is complex. Detailed knowledge of the flow resistance, or viscosity, of grease is needed in attacking practical and theoretical problems. Both accurate measurements and correct mathematical interpretations are necessary to index viscosity.

The most common instrument for measuring viscosity is the capillary viscometer. Many different viscometers have been successfully used. Measurement of flow rates and pressures through capillaries is not difficult.

Interpretation of results, however, is not easy. Four ways of mathematically interpreting observed flow rates and pressures have been devised. Which should be used to define the flow of grease for both practical and theo-

retical purposes? Does any provide results that can be used generally? The questions come easy; the answers come hard. To answer them, a review is needed of what we know about measuring flow rates and pressures in capillary viscometers and about interpreting these measurements.

VISCOMETERS

At a given temperature, three variables affect the flow of grease through capillaries: rate of flow, pressure necessary to maintain flow, and dimensions of the capillaries. These variables are measured in different ways in different viscometers.

Viscometers of Arveson² and the ASTM¹ allow pressure measurements at constant flow rates through capillaries of several radii. The grease is forced through the capillary by a floating piston actuated by a hydraulic system.

Viscometers of Singleterry,¹⁵ Blott,⁴ and Marusov¹¹

provide for reciprocating movement of the grease through the capillary. These instruments have the advantage of measuring changes in viscosity occurring because of changes in the grease during prolonged shearing. In the first two, flow is timed while a constant pressure is applied; in the last, the flow rate is fixed and the pressure is measured.

These and other viscometers^{3,5,8,15} lead to values of pressures and flow rates in capillaries of known dimensions. Corrections peculiar to each viscometer must be made to provide sound data for interpretations.

TYPES OF FLOW

Flow equations are usually based on some ideal system. Two systems may be applicable: one based on a Newtonian liquid and one based on a Bingham plastic.

If capillary flow is completely laminar, the ratio of the shearing stress, F , to the rate of shear, dv/dr , equals a constant, called viscosity, η . According to Newton's equation:

$$\eta = \frac{F}{dv/dr}$$

where v is the linear velocity and r is the radius of the capillary. For Newtonian flow in a capillary, Poiseuille's integration¹⁶ applies,

$$\eta = \frac{Pr/2L}{4Q/\pi r^3}$$

where P is the pressure; r , the radius of the capillary; L , the length of the capillary; and Q , the flow rate. In any one viscometer, where r and L are constant,

$$\eta = K \frac{P}{Q}$$

where the constants are collected to give $K = \pi r^4/8L$.

Many materials do not flow completely laminarily. One such system is the ideal plastic defined by Bingham's equation:

$$\eta = \frac{F - f}{dv/dr}$$

in which f is the yield value. A Bingham plastic totally resists flow as force is applied until a critical force is exceeded. Under larger forces, the flow is laminar. Applying this equation to flow in capillaries requires Buckingham's integration⁸ of the Bingham equation,

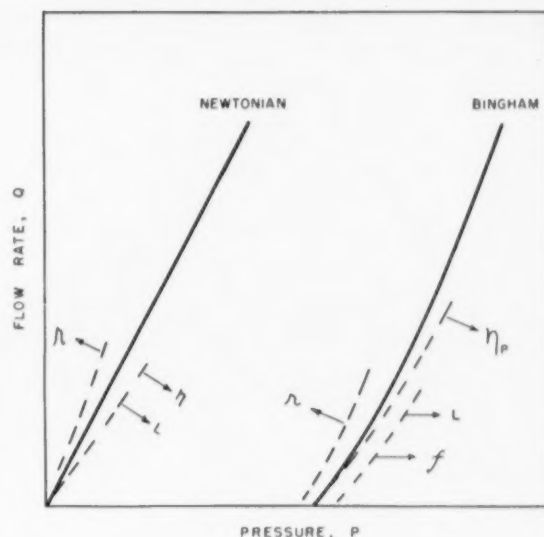
$$Q = \frac{\pi r^4}{8L\eta p} (P - \frac{4py}{3} + \frac{p^2y}{3P^3})$$

The limiting viscosity, η_p , and yield value, $f = \phi(py)$, are presumed constant for any one material.

The left curve in Figure 1 shows the linear relationship obtained for Newtonian liquids. The right curve shows the typical relationship for a Bingham plastic. Buckingham's equation allows three situations as the applied force is increased: below the yield pressure, py , no flow occurs; above the yield pressure but below the straight-line portion of the curve, plug and laminar flow occurs; at larger forces, complete laminar flow occurs. Arrows indicate the changes that occur as r , L , η , η_p , and f increase.

Lubricating materials differing widely in flow properties are included under the term "grease." Some flow like

Figure 1
TYPES OF FLOW



a Newtonian liquid and some like a Bingham plastic. Furthermore, many greases change during flow,²⁰ becoming less viscous (thixotropic) or more viscous (rheopectic, dilatant), and further complicate interpretation of the nature of the flow.

INTERPRETATIONS

The literature reveals four mathematical ways to describe the flow of grease. If flow is assumed to be related to that of a Newtonian liquid, two ways can be used: apparent viscosity and viscosity at the wall. If flow is assumed to be more like that of a Bingham plastic, a method based on the Buckingham integration can be used. If flow is considered to be a rate process, an equation can be derived from flow data through the use of Eyring's relaxation theory for viscous flow.⁹ These treatments range from the empirical to the theoretical.

Apparent Viscosity

The commonest means for obtaining apparent viscosity is the ASTM method.¹ Shearing stress, F , is calculated by the equation:

$$F = Pr/2L$$

Because the method fixes the ratio, r/L , for all capillaries used, this equation reduces to $F = PC$. Shear rate, S , (or dv/dr), is calculated by the equation for a Newtonian liquid:

$$S = Q \frac{4}{\pi r^3}$$

A plot of F and S derived from P and Q is shown in Figure 2. For Newtonian liquids, the family of straight lines on the P - Q plot becomes one straight line on the F - S plot. For most greases, the P - Q curves in different capillaries are not straight lines and do not extrapolate to the origin. They appear to become a single curve on an F - S plot. The range of flow rates in the ASTM method is too narrow to reveal deviations from the average line. However, when even a wider range is used, as shown

Figure 2

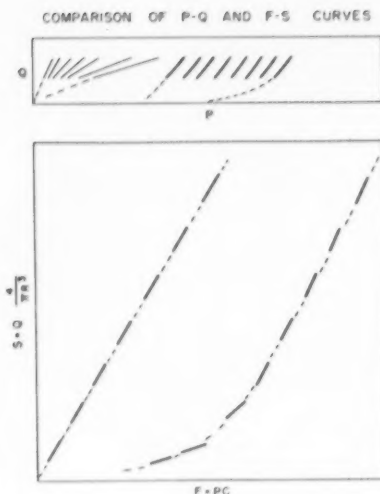


Figure 3

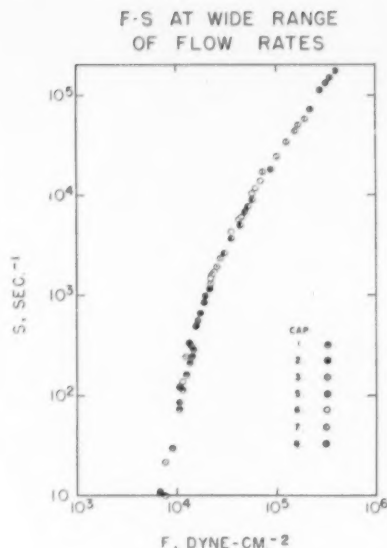
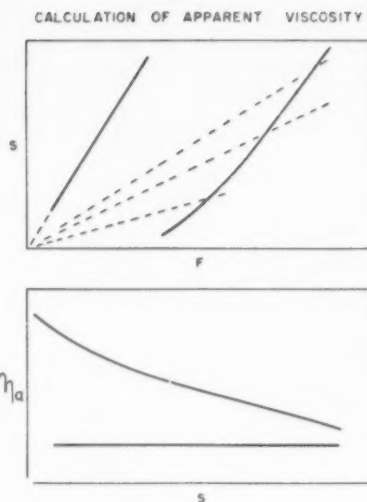


Figure 4



in Figure 3, the data still appear to be on a single curve. These data were obtained in capillaries of constant ratio, r/L .

The method calculates viscosity from F and S . Because F/S is not constant, the calculated number is called apparent viscosity and is represented by a locus of points on a curve of apparent viscosity, η_a , versus shear rate, S . Figure 4 shows a plot of these values for a Newtonian liquid and a typical grease.

Although several errors occur in measured pressures, the ASTM method makes no provision for correcting them. Generally recognized sources of errors are temperature effects, kinetic-energy effects, friction in the cylinder, convergence at the capillary opening, and orientation effects. Temperature corrections can only be made by analogy with Newtonian-liquid systems. Kinetic-energy corrections are usually insignificant in this viscometer. Friction, convergence, and orientation can be corrected for some greases. The magnitude of all these errors varies with each grease.

Figure 5 relates η_a to S for a grease and shows the results of correcting the data. The upper curve shows data obtained as prescribed by the ASTM method. The middle curve shows the same data corrected for convergence and friction by subtracting from the observed pressure that pressure required to force grease through 0.005-inch plates having holes the same diameter as each capillary. The lower curve shows data corrected also for orientation by subtracting from the observed pressure that pressure obtained from a shorter capillary ($L/D = 12/1$). For a grease like that shown in Figure 5, Singleterry¹⁸ claims that orientation occurs in 1 to 2 diameters. These corrections do not alter the shape of the η_a curve but do reduce the values of η_a . Corrected data should be used in comparing ways of interpreting grease flow.

Although the ASTM method may lead to a single, reproducible curve for a grease, F/S is by no means a

proper representation of this curve. The entire spectrum of F/S values is needed to define grease flow.

Viscosity at the Wall

Another mathematical treatment, first applied to greases by Blott⁴ and later by Marusov,¹¹ defines shear stress and shear rate at the wall. The mathematics was developed originally by Rabinowitsch¹⁷ and further by Mooney.¹⁴ The equation for viscosity at the wall, η_w , is:

$$\eta_w = \frac{F}{3Q/\pi r^3 + F d(Q/\pi r^3)/dF}$$

where, by analogy with Poiseuille's equation, shear rate at the wall, S_w , is $3Q/\pi r^3 + F d(Q/\pi r^3)/dF$.

The method of calculation outlines the three steps shown graphically in Figure 6.

Plot flow rate, Q , versus shear stress, F ($= Pr/2L$), and determine the slope, dQ/dF .

Plot shear rate at the wall, S_w , versus F .

Determine viscosity at the wall, η_w , and plot η_w versus S_w .

To compare η_w with η_a , published data from three sources were used, as shown in Figure 7. Curve a is from Arveson²; the points are recalculated from this data to get values for η_w . Curve b is the η_a curve calculated from the data of Blott.⁴ Curve c is the η_a curve calculated from the data of Marusov.¹¹ The coincidence of these points and curves show the close resemblance between η_w and η_a .

Buckingham Equation

Buckingham's equation may be written

$$Q = \frac{\pi r^4 m}{8 L} \left(P - \frac{4p_y}{3} + \frac{p_y^3}{3P^3} \right)$$

where m is the mobility ($=1/\eta_v$). Caldwell and Babbitt⁷ reduced the quantity in parentheses to a dimensionless term that is a function only of the relative extent of plug flow:

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Figure 5

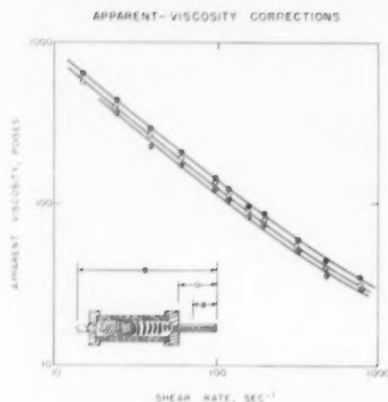
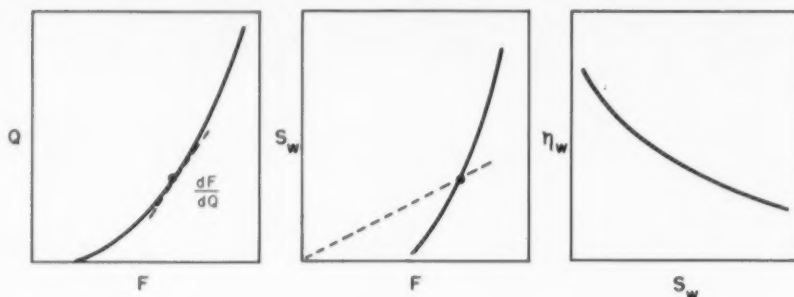


Figure 6

CALCULATION OF VISCOSITY AT THE WALL



$$Q = \frac{\pi r^4 m P}{8 L} \left(1 - \frac{4 p_y}{3 P} + \frac{p_y^3}{3 P^3} \right)$$

McMillen¹² solved the equation in terms of two dimensionless quantities:

$$c = b/r$$

$$\alpha = \frac{c^4 - 4c + 3}{12c}$$

where b is the radius of the unsheared plug. He reduced these equations to graphs of dimensionless constants that make solution possible. The McMillen equations for the calculation are:

$$f = \frac{Prc}{2L}$$

$$m = \frac{Q}{\pi r^4 f \alpha}$$

$$\eta_p = 1/m = (P/Q) \frac{\pi r^4 c \alpha}{2 L}$$

Singleterry and Stone¹⁸ made further use of graphs and applied the McMillen equations to greases.

Calculation of f and m by means of the Buckingham equation involves 6 steps outlined in Figure 8.

Plot flow rate, Q , in one capillary, versus pressure, P .

Select a ratio P_2/P_1 ($= c_1/c_2$), using the largest experimental range possible.

Read corresponding values for Q_1 and Q_2 from the P - Q plot.

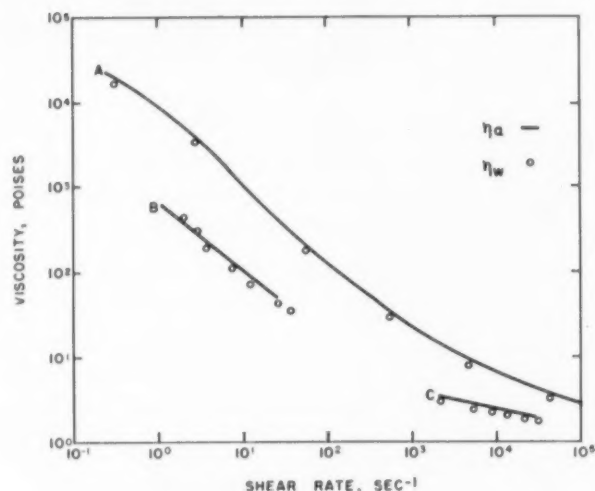
Determine c_2 from a plot of α_1/α_2 ($= Q_1/Q_2$) versus c_2 .

Determine α_1 and α_2 from c_1 and c_2 by a plot of α versus c .

Calculate f and m from the McMillen equations.

The curve for the Buckingham equation can be determined.

Applications of the McMillen equations to grease have been made. Singleterry,¹⁸ using only one capillary, found good agreement between P - Q data and the Buckingham equation. P - Q data on three greases were obtained from an ASTM viscometer by the authors. Pressures were corrected. Calculations of f and m were made for each grease from data from four capillaries. The results are shown in Table I. Grease A is similar to that used by

Figure 7
COMPARISON OF APPARENT VISCOSITY
AND VISCOSITY AT THE WALLTABLE I
Yield Value and Limiting Viscosity at 77° F

Grease	A		B		C	
Oil Vis., SSU 100° F	70		300		800	
NLGI Grade	0		2		1.5	
ASTM	f	η_p	f	η_p	f	η_p
Capillary	dyne/cm ²	poises	dyne/cm ²	poises	dyne/cm ²	poises
1	5400	22	8600	..	3800	62
2	8100	21	5400	27
3	9100	15	5400	22
5	6900	3	8900	13	10,000	7

Singleterry. The values for f were nearly constant for greases A and B but not for C. In all greases the values of η_p were not constant. An attempt was made to apply the Buckingham equation constants obtained from one

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capillary to the calculation of the Buckingham line for a capillary of different radius. In Figure 9 the dotted line, *c*, represents the Buckingham equation calculated from the data of capillary *a* which should correspond to line *b*. Although these greases appear to have flow curves resembling the P-Q curves for Bingham plastics, they may actually demand mathematics that are more complex.

CONCLUSION

Regardless of the mathematics applied, flow-pressure data is needed to solve four kinds of problems: for specifying greases for a particular problem in lubrication; for empirically correlating flow in capillaries and in practice; for calculating pressures and flow rates under specified conditions; and for understanding grease structure. The choice of mathematics depends upon the problem.

Specifications usually depend upon apparent viscosity. Viscosity at the wall could be used as well. Both require the use of a full curve to specify the grease. If further improvements can be made in the Buckingham equation, the use of two constants, *f* and *m*, holds great promise.

Empirical correlations have been found between apparent viscosity and other variables.^{2,3} Correlations using η_w , *f*, and *m* could be equally useful.

No way of calculating flow rates and pressures in pipes has proved successful. Dispensing qualifications¹² that have been worked out are empirical and are based on η_w . Weltmann¹⁹ and Hedstrom¹⁸ have both successfully used the Buckingham equation to predict the flow of plastic systems similar to greases.

Fundamental properties of greases have not been correlated with grease flow. Such thickener properties as size, shape, and particle interaction are of obvious importance. Fyring's equation contains parameters that might be related to some of them. This approach holds great promise and justifies further study.

There remains a need to improve our knowledge of the nature of the flow of greases. Although each of several mathematical ways discussed may be applied, no way rigorously defines grease flow.

Figure 8
CALCULATION OF BUCKINGHAM CONSTANTS

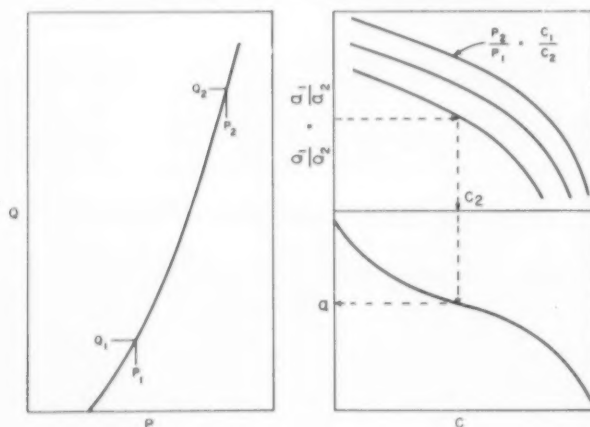
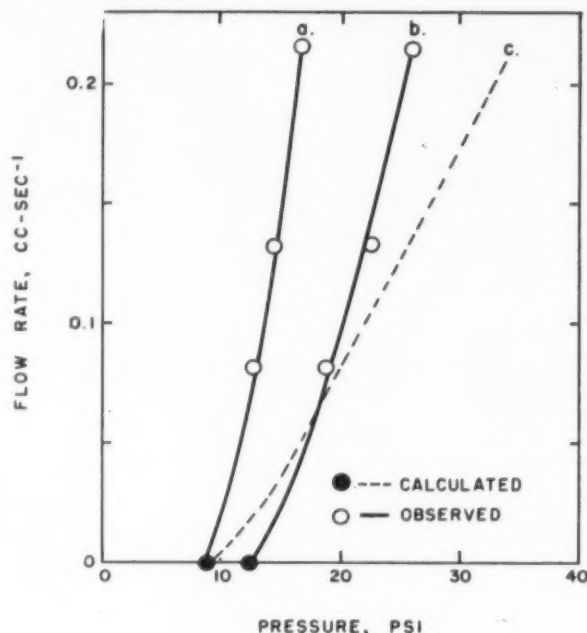


Figure 9
COMPARISON OF FLOW DATA
WITH BUCKINGHAM EQUATION



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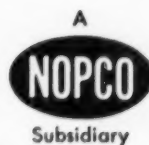
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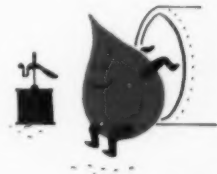
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RHEOLOGICAL MEASUREMENTS and Their Application to Pipe-Flow Properties



By RUTH N. WELTMANN, NACA Lewis Flight Laboratory, Cleveland, Ohio

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INTRODUCTION

OILS AND GREASES which can be of Newtonian and non-Newtonian flow behavior frequently have to be passed through pipe lines for transport and lubrication purposes. The pressure losses suffered during the passage of these materials will determine the design of the pipe lines. Since the flow characteristics of the materials greatly affect the pressure losses, the knowledge of these properties is important and their measurements with a concentric-cylinder rotational viscometer are discussed.

The flow property of a Newtonian liquid is constant under all flow conditions at constant temperature and pressure, while the flow properties of non-Newtonian materials are shear-dependent and for many materials also time-dependent. Therefore, the flow properties of non-Newtonian materials can only be obtained from carefully timed and programmed curves of rate of shear as a function of shearing stress, which must be measured with a properly designed instrument. One such an instrument is the rotational viscometer built at NACA. Some of the design features of this instrument are briefly described and flow curves of two lubricants obtained with this viscometer are presented.

The pressure loss of a Newtonian fluid when passing through a pipe line is determined from the friction factor which is a unique function of the Reynolds number, and can be obtained from the well-known friction diagram, Ref. 1, if the flow velocity, pipe diameter and the viscosity are known. For non-Newtonian materials a more general friction diagram, Refs. 2 and 3, has to be used, from which again a friction factor is obtained. Since two flow properties are required to describe the flow of a non-Newtonian material at any one flow condition, the friction factor is not any more a unique function of the Reynolds number but is also a function of a second dimensionless parameter which is either the plasticity or the structure number depending on the flow properties

of the non-Newtonian material. Thus, the pressure loss of a non-Newtonian material in a pipe line can be determined if the flow velocity, the pipe diameter, and two flow properties at the flow condition in the pipe line are known. Sample calculations of pressure losses are presented for the flow of two non-Newtonian lubricants in various pipe line systems.

SYMBOLS

The following symbols are used in this paper:

- C rate of shear correlation factor
- D diameter of pipe, [L]
- f yield value, $[ML^{-1}T^{-2}]$
- G rate of shear, $[T^{-1}]$
- h height of annulus, [L]
- K constant in equation (5)
- k instrument constant, $k = k_1/k_2$
- k_1 instrument constant in equation (1)
- k_2 instrument constant in equation (2)
- k_3 instrument constant in equation (4)
- L length of pipe, [L]
- N structure number
- Pl plasticity number, $Pl = fD/Uv$
- Δp pressure loss, $[ML^{-1}T^{-2}]$
- Q volume flow rate, $[L^3T^{-1}]$
- R_i radius of inner cylinder, [L]
- R_o radius of outer cylinder, [L]
- Re Reynolds number, $Re = Dv\rho/\text{viscosity}$
- r any radius between R_o and R_i , [L]
- T torque, $[ML^2T^{-2}]$
- U plastic viscosity, $[ML^{-1}T^{-1}]$
- v mean velocity, $[LT^{-1}]$ (from flow rate)
- η apparent viscosity, $[ML^{-1}T^{-1}]$
- μ Newtonian viscosity, $[ML^{-1}T^{-1}]$
- ρ density, $[ML^{-3}]$
- τ shearing stress, $[ML^{-1}T^{-2}]$
- φ friction factor
- ω angular velocity, $[T^{-1}]$

Subscripts:

- O intercept
- G shear dependent
- T tangent
- t time dependent

FLOW PROPERTIES

The flow properties of any type of material can best be determined from a measurement of a flow curve, which is a curve of rate of shear against shearing stress. Flow curves representing different types of materials are shown in Table I together with the respective flow properties. Flow curves are measured by shearing the material in a viscometer under defined shear conditions and at a constant temperature. The two most popular viscometers are the capillary tube and the concentric-cylinder rotational viscometers. In this paper only the latter will be discussed since it is well suited for the measurement of flow curves of all types of materials, be they Newtonian liquids, shear-dependent or shear- and time-dependent non-Newtonian materials.

In a concentric-cylinder rotational viscometer two cylinders are rotated with respect to each other and the sample is shear in the confined annular space between the two cylinders. Measurements are made of the angular velocity ω applied to the rotating cylinder and of the torque T exerted on the stationary cylinder by the sample. Turbulent flow in the sample must be avoided.

Under laminar flow conditions, the rate of shear in a concentric-cylinder rotational viscometer is proportional to the measured angular velocity and is,

$$G = \frac{2\omega}{r^2 \left(\frac{1}{R_b^2} - \frac{1}{R_c^2} \right) C} \quad (1)$$

and the shearing stress is proportional to the measured torque and is

$$\tau = \frac{T}{2\pi r^2 h} \quad (2)$$

Both G and τ are inversely proportional to r^2 and thus increase across the annular space from R_c to R_b . The factor C equals 1 for shear-independent materials. For shear-dependent materials, C depends on their degree of shear-dependency and is then also a function of r , Ref. 4. For a viscometer with a small annular width, it is suggested to use the midpoint radius, $r = (R_c + R_b)/2$ for calculating G , τ , and C , Ref. 2. Then $G = \omega/k_1 C$ and $\tau = T/k_2$, where,

$$k_1 = \frac{(R_c + R_b)^2}{8} \left(\frac{1}{R_b^2} - \frac{1}{R_c^2} \right) \text{ and } k_2 = \pi h (R_b + R_c)^2.$$

After a flow curve is obtained, the flow type of the material and its flow properties can be determined.

Newtonian. The flow curve of a Newtonian liquid is a straight line, Table I. Therefore, one flow property, the viscosity μ describes a Newtonian liquid. Since the Newtonian viscosity is shear-independent and constant for any r across the annular space between R_c and R_b , the correlation factor C is equal to 1. When the material is definitely known to be Newtonian, a one-point measurement is sufficient to determine its viscosity; however, many materials which are believed to be Newtonian are found to be non-Newtonian at higher rates of shear or

at lower temperatures.

Bingham Plastic. The flow curve of a Bingham plastic, Ref. 5, Table I, is also a straight line but only after a certain minimum shearing stress τ_0 is applied. At any shearing stress below τ_0 , no flow occurs and the sample behaves like a solid, while at any shearing stress above τ_0 the sample will flow. Therefore, two flow properties, the plastic viscosity U and the yield value f are required to describe the flow of a Bingham plastic. The plastic viscosity U in a rotational viscometer, for $k = k_1/k_2$, is

$$U = \frac{\tau - \tau_0}{G} = k \frac{(T - T_0)}{\omega} \quad (3)$$

Since the plastic viscosity U is shear-independent and constant for any r across the annular space between R_c and R_b , at any $\tau \geq f$, the factor C again is equal to 1. The yield value f in a rotational viscometer is

$$f = \tau_0 = \frac{r^2 \left(\frac{1}{R_b^2} - \frac{1}{R_c^2} \right) T_0 \left(\frac{1}{R_b^2} - \frac{1}{R_c^2} \right)}{2 \ln \left(\frac{R_c}{R_b} \right) 4\pi h \ln \left(\frac{R_c}{R_b} \right)} = k_3 T_0 \quad (4)$$

Although τ_0 is a function of r , the yield value f is shear-independent and constant across the annulus.

Pseudoplastic and Dilatant. The flow curves of a pseudoplastic and a dilatant material, Table I, are nonlinear. They can frequently be represented by the empirical relationship, Ref. 6,

$$G \propto \tau^N \quad (5)$$






At least two flow properties, the apparent viscosity η_0 and the structure number N , are required to describe these materials. The apparent viscosity η_0 is always shear-dependent which is indicated by the subscript G , and is defined as

$$\eta_0 = \frac{\tau}{G} = k \frac{TC}{\omega} \quad (6)$$

where C is larger than 1 for a pseudoplastic and smaller than 1 for a dilatant material and depends on R_c , R_b , and N , Ref. 4. The factor C can be made negligibly small by proper design of the viscometer. For instance, C will be less than 1.05, for N up to 7, if the cylinders are designed so that $R_b/R_c \geq 0.925$, Ref. 2. The structure number N

Table I

FLOW CURVES AND FLOW PROPERTIES

MATERIAL	NEWTONIAN	BINGHAM PLASTIC	PSEUDO-PLASTIC	DILATANT	TIME-DEPENDENT
FLOW CURVES					
FLOW CURVE EQUATIONS	$G = \frac{1}{\mu} \tau$	$G = \frac{1}{\mu} (\tau - \tau_0)$	$G \propto \tau^N$	$G \propto \tau^N$	
ROTATIONAL VISCOMETER $G = \omega/k_1 C$ $\tau = T/k_2$	$C = 1.00$	$C = 1.00$	$C > 1.00$	$C < 1.00$	$C \neq 1.00$
FLOW PROPERTIES	μ	U f	η_0 N or $N_0 > 1$	η_0 N or $N_0 < 1$	η_0, t or η_0, t τ_0, t or τ_0, t
ROTATIONAL VISCOMETER EQUATIONS	$\mu = k \frac{T}{\omega}$	$U = k \frac{T - T_0}{\omega}$ $f = k_3 T_0$	$\eta_0 = k \frac{T \cdot C}{\omega}$ $N = \frac{1 \log \frac{\omega}{\omega_0}}{1 \log \frac{\tau}{\tau_0}}$ or $N_0 = \frac{1 \log \frac{\omega}{\omega_0}}{1 \log \frac{\tau}{\tau_0}}$		

is obtained by plotting $\log G$ against $\log \tau$. If N is shear-independent, this plot is linear and N is the slope of this line. In many materials N_G is shear-dependent and the plot of $\log G$ against $\log \tau$ is not linear. In the case where both flow properties are shear-dependent, η_G and N_G have to be obtained for the rate of shear to which the material is subjected during its industrial application. The structure number N is larger than 1 for pseudoplastic materials and is smaller than 1 for dilatant materials.

Time-dependent. The three types of non-Newtonian materials described are frequently not only shear-dependent but also time-dependent. This can be evidenced by the difference between two flow curve measurements, (where one is obtained at increasing rates of shear and the other subsequently at decreasing rates of shear) since the flow properties of time-dependent materials change with the history of sample treatment. A flow curve of a time-dependent material is shown in Table I. The flow curve A measured at increasing rates of shear differs from curves B and C measured at decreasing rates of shear. The pronounced difference between the two curves A and B indicates that the decrease in flow properties is caused not only by an increase in shear, but is also time-dependent. A different flow curve C is obtained indicating a further decrease in flow structure when the material is sheared for a certain time at the maximum rate of shear before the rate of shear is decreased for the flow curve measurement. Such a behavior of a time-dependent material indicates either permanent breakdown or thixotropic behavior. A thixotropic material will recover its original structure when left undisturbed for a period of time. Thus, a second flow curve measurement is required to classify the flow behavior of such a material. Meaningful flow curves of time-dependent materials can be obtained only if the measurements are made at a constant rate and according to a programmed sequence. Flow properties analogous to the ones described for only shear-dependent non-Newtonian materials can then be determined for different shear and time conditions.

FLOW CURVES OF LUBRICANTS

The flow curves of two lubricants were obtained with a concentric-cylinder rotational viscometer, Figure 1, which was built at NACA. This viscometer, Ref. 7, permits the automatic recording of flow curves at constant acceleration and in accordance with a predetermined program. The outer cylinder is rotated by a motor in accordance with a preset program, while the torque is measured with a force transducer on the inner stationary cylinder. Angular speed and torque are recorded on an X-Y recorder. The two cylinders are so designed that the rate of shear correlation factor C is approximately equal to 1.00 for most materials and thus can be neglected. The viscometer can measure materials ranging in viscosity from 0.05 to 20,000 poises. The maximum rate of shear is 4000 sec^{-1} and the shearing stress ranges from 50 to 250,000 dynes per square centimeter. A special piston pump is provided to make possible the proper injection of grease-like materials in the viscometer.

Figures 2, 3, and 4 are flow curve records of two auto chassis lubrication greases which are designated as grease T-M and grease S. The records were taken by increasing

the rate of shear up to a maximum rate of shear (up-curve) and by decreasing the rate of shear to zero rate of shear (down-curve) immediately thereafter. The up- and down-curves were recorded in 60 seconds under

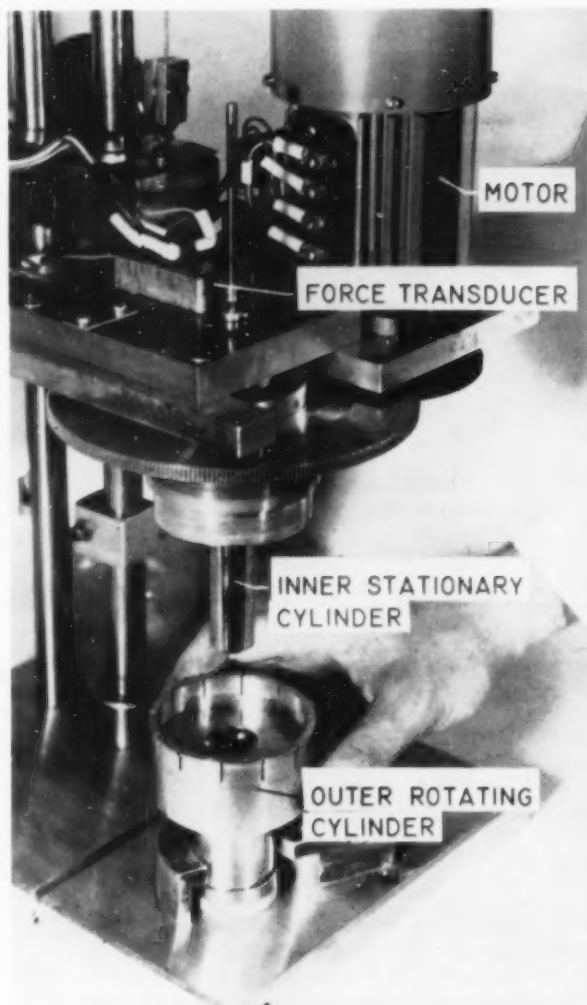


Figure 1. NACA rotational viscometer.

constant acceleration and with the sample in a constant temperature bath of 30° C. The concentric cylinders used had a ratio of $R_b/R_c = 0.925$. Both greases were treated alike prior to the measurement.

Grease T-M. Grease T-M, Figure 2, was measured to a maximum rate of shear of about 1150 sec^{-1} . The difference between the up- and down-curves of grease T-M is small indicating that this grease is relatively time-independent. If treated as a pseudoplastic (eq. 5) the structure number is $N = 1.3$ for $G \geq 40 \text{ sec}^{-1}$ for both curves, and N increases for lower rates of shear. The rate of shear correlation factor C can be neglected since the error introduced by doing so is very much less than 1 per cent. The apparent viscosity η_G changes with the rate of shear.

Grease S. Two flow curves, Figures 3 and 4, were recorded for grease S. They represent measurements to

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$G = 1200$ and $G = 250 \text{ sec}^{-1}$, respectively. The up- and down-curves in both figures differ greatly indicating that grease S is time-dependent. Although a constant temperature bath was used, part of the difference might be caused by a temperature rise during shear. The up-curves if treated as pseudoplastics have structure numbers of $N = 4.0$ and $N = 3.0$, when measured in 30 seconds to $G = 1200$ and $G = 250 \text{ sec}^{-1}$, respectively. The N of both down-curves is shear-dependent. Even for $N = 4.0$, C can be neglected, since the error introduced by doing so is only about 1 per cent. The apparent viscosity of this material $\eta_{a,t}$ is shear- and time-dependent. The down curves resemble those of Bingham plastics with different U and f , depending on the maximum applied rate and time of shear. Since this grease is time dependent the flow properties obtained from a down-curve will be assumed to describe the flow condition that the material has after being subjected to the applied maximum rate and time of shear which is either 1200 sec^{-1} in 30 seconds, Figure 3, or 250 sec^{-1} in 30 seconds, Figure 4.

PRESSURE LOSS IN PIPE FLOW

The pressure loss in a pipe line, when considering only the straight portion is given as

$$\Delta p = \rho v^2 L \frac{\phi}{2 D} \quad (7)$$

The friction factor ϕ can be obtained for Newtonian, Bingham plastic, pseudoplastic, and dilatant materials from the generalized friction diagram, as shown in Figure 5, Refs. 2 and 3. In this diagram ϕ is plotted as a function of the Reynolds number Re , where

$$Re = Dv\rho/\text{viscosity} \quad (8)$$

Newtonian. For a Newtonian liquid the viscosity in equation 8 is the Newtonian viscosity μ . The friction

diagram for Newtonian fluids is well known, Ref. 1, and is represented by curves a and b in Figure 5. Since ϕ is a function of Re only, the pressure loss caused by transferring a Newtonian material through a pipe line of given pipe diameter and at a given velocity is uniquely determined by the viscosity μ of the liquid.

Non-Newtonian. The flow properties of non-Newtonian materials are used in an analogous manner to determine the pressure loss in a pipe line caused by their passage. For non-Newtonian materials the generalized friction diagram, Figure 5, gives ϕ also as a function of Re but along different lines of constant plasticity and structure numbers, Refs. 2, 3 and 8.

For a Bingham plastic the friction factor is found from Figure 5 at the intersection of the Reynolds and plasticity numbers where the plastic viscosity U is used in equation 8 to determine Re , and Pl is a function of plastic viscosity and yield value and is, Ref. 8,

$$Pl = fD/Uv \quad (9)$$

The two flow properties of a Bingham plastic, U and f , are independent of the rate of shear in the pipe line and thus it is not necessary to know this shear rate to calculate the pressure loss caused by the passage of these materials.

For a pseudoplastic and dilatant material the friction factor is found also from Figure 5 at the intersection of the Reynolds and structure numbers where the apparent viscosity η_a is used in equation 8 to determine Re and has to be obtained for the rate of shear to which the material is subjected in the pipe line. If N is also shear dependent, an N_a has to be obtained for the same rate of shear. Therefore, to calculate the pressure losses caused by the passage of these materials it is necessary to know the particular shear rate in the pipe line, which for a material represented by equation 5 is, Refs. 2 and 4.

Figure 2. Flow curves of grease T.M.

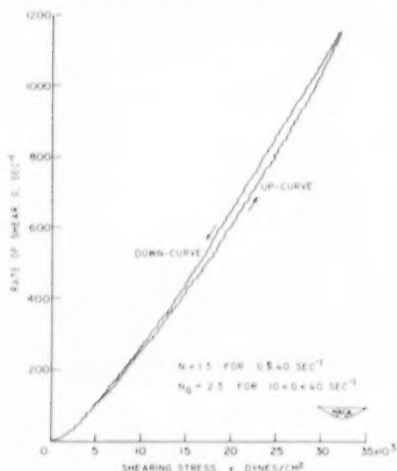


Figure 3. Flow curves of grease S, measured up to $G=1100 \text{ sec}^{-1}$ in 30 seconds.

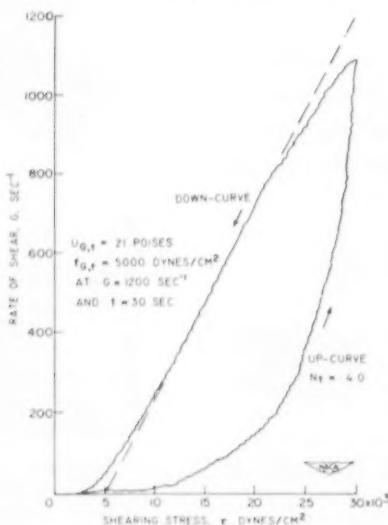
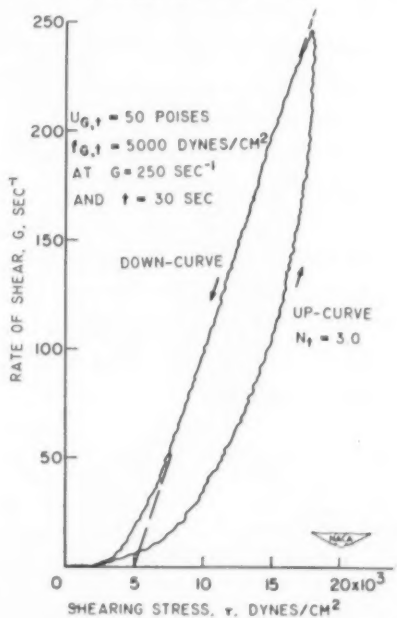
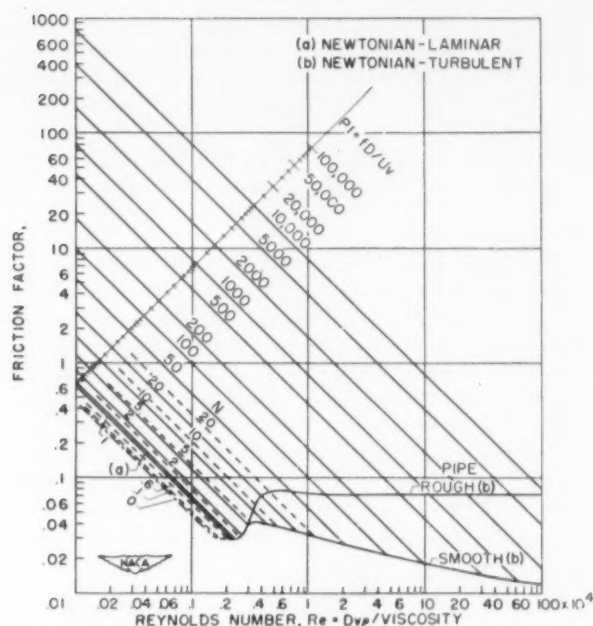


Figure 4. Flow curves of grease S, measured up to $G=250 \text{ sec}^{-1}$ in 30 seconds.





$$G_{\text{pipe}} = \frac{2v(N+3)}{D} \quad (10)$$

It is seen from Figure 5 that at Reynolds numbers equal or higher to those, where the lines of constant Pl and N intersect the friction curves for Newtonian liquids, only U and η determine the friction factor. Whenever the apparent viscosity is to be determined at a high shear rate, an apparent viscosity extrapolated to infinite rate of shear can be used in the Reynolds number. This viscosity can be found from a plot of reciprocal rate of shear against apparent viscosity, Ref. 9, by extrapolation to $1/G = 0$.

Figure 5. Left, Friction diagram for Newtonian and non-Newtonian flow in pipe lines.

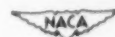
PRESSURE LOSS CALCULATIONS

In industrial application lubrication greases are often made to flow through circular conduits of several feet in length and ranging from 1/8 inch to 1.0 inch in diameter. To calculate the required pressure, for obtaining a desired flow rate or velocity necessitates the prediction of the pressure losses in these pipe lines when passing the lubricant. Pressure loss calculations are presented in Table II for the two lubricants whose flow curves are given in Figures 2, 3, and 4. It is assumed for the calculations that the pipe dimensions and flow rates are given and that the density of the materials is equal to 1 gram per cubic centimeter.

Since lubricant grease T-M is a pseudoplastic material, the rates of shear in the pipe line under operating conditions were calculated by using equation 10 after N was obtained from the flow curve, Figure 2. Then η_a was obtained for these rates of shear from the flow curve to calculate Re . The friction factor was found from Figure 5 and Δp was calculated from equation 7. In some cases the pressure loss can be more rapidly approximated by using a tangent on the flow curve of a pseudoplastic material and by treating the pseudoplastic as if it were a Bingham plastic. This was done for grease T-M to evaluate the validity of such an approximation. Using this "tangent" method a plastic viscosity U and a yield value f were obtained from the slope and intercept of the tangent to the flow curve which was constructed at a rate of shear $G_T = 8v/D$. This rate of shear G_T approximates the rate of shear in the pipe line, equation 10, closely enough for the tangent method and has the advantage that N is not required for its calculation. The parameters Re and Pl were obtained from U and f and the friction factor is found from Figure 5 and Δp from equation 7. As expected the pressure losses calculated by

Table II

PRESSURE-LOSS CALCULATIONS FOR TWO LUBRICATION GREASES



GREASE	Q , CC SEC	D , IN.	L/D	v , CM SEC	G_T , SEC ⁻¹	G_{PIPE} , SEC ⁻¹	N	η_G , POISES	U , POISES	f , DYNES CM ²	Re	Pl	ϕ	Δp , PSI	METHOD
T-M	4.1	1/4	1000	13		180	1.3	42.2			0.194		350	430	PSEUDO- PLASTIC
	32.3	1/2	1000	26		180	1.3	42.2			0.780		88	430	
	136	1/2	1000	110		750	1.3	30.3			4.60		15	1300	
	560	1.0	1000	110		370	1.3	36.0			7.70		8.7	760	
	1120	1.0	1000	220		750	1.3	30.3			18.4		3.7	1280	
T-M	4.1	1/4	1000	13	160				29.0	2000	0.283	3.4	340	420	TANGENT
	32.3	1/2	1000	26	160				29.0	2000	1.13	3.4	87	430	
	136	1/2	1000	110	700				24.0	4500	5.82	2.2	15	1300	
	560	1.0	1000	110	350				28.5	2300	9.70	1.8	8.7	760	
	1120	1.0	1000	220	700				24.0	4500	23.3	2.2	3.7	1280	
S	4.1	1/4	1000	13		250	3.0		50.0	5000	0.165	4.9	720	880	TIME- DEPEND- ENT
	32.3	1/2	1000	26		250	3.0		50.0	5000	0.660	4.9	180	880	
	136	1/2	1000	110		1200	4.0		21.0	5000	6.50	2.7	15	1300	
	1120	1.0	1000	220		1200	4.0		21.0	5000	26.0	2.7	3.7	1280	

use of the tangent method check closely with the ones obtained by using the two pseudoplastic flow properties.

For time-dependent materials, pressure losses cannot be calculated unless the flow properties at the rate and time of shear in the pipe line are known. Therefore, for the sample calculations of pressure losses for grease S, Table II, which according to its flow curves, Figures 3 and 4, is time-dependent, various assumptions had to be made. It was assumed that the mean rates of shear in the pipe lines are given by equation 10, when using the N of the up-curves, that the mean time of shear is 30 seconds, and that the flow properties of the down-curves are representative for the flow condition of this grease at those rates and time of shear. Then, the parameters Re and Pl were obtained from $U_{a,t}$ and $f_{a,t}$ of the respective down-curve, ϕ was found from Figure 5 and Δp was calculated from equation 7. However, until pressure losses of time-dependent materials in pipe lines are analyzed by experiment, the above assumptions cannot be verified. Hence these pressure loss calculations can be considered to be approximations at best.

For a Newtonian material in laminar flow the pressure loss in a pipe line increases linearly with the velocity and is inversely proportional to the pipe line diameter for a constant L/D . From Table II it is seen that for a pseudoplastic material such as grease T-M, the pressure loss increases slower than linearly with the velocity and that the inverse ratio of two pressure losses when calculated for a constant flow velocity is less than the ratio of the diameters of the pipe lines for which the pressure losses were obtained. Thus, the behavior of a non-Newtonian material when flowing through a pipe line in laminar flow is quite different from that of a Newtonian liquid.

CONCLUDING REMARKS

The discussion in this paper shows how to obtain flow properties for various types of non-Newtonian ma-

terials from flow-curve measurements with a concentric-cylinder rotational viscometer and how to use these flow properties to calculate pressure losses in straight pipe lines due to the flow of Bingham plastic, pseudoplastic, and dilatant materials.

Flow curves of two lubrication auto chassis greases are presented. They indicate that one grease is a pseudoplastic material while the other one shows a time-dependent flow behavior.

Pressure losses caused by the transfer of the pseudoplastic grease are calculated for various pipe line systems.

Calculations of pressure losses in pipe lines due to the transfer of the time-dependent grease are attempted. However, since experimental pressure loss data are not available for time-dependent materials these calculations had to be based on various assumptions.

The pressure losses calculated for the flow of the non-Newtonian lubricants indicate that the laminar flow behavior of non-Newtonian materials in a pipe line is quite different from that of a Newtonian liquid.

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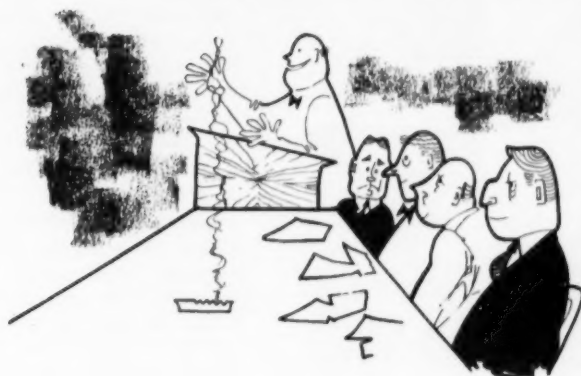


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QUESTION AND ANSWER SESSION



Lubricating Greases for Modern Farm Machinery

Editor's Note—The NLGI panel discussion on lubricating greases for modern farm machinery presented at the Annual Meeting in Chicago, October, 1955, resulted in a lively question and answer session conducted by panel chairman M. L. Carter. Reproduced here is an actual transcript from this discourse, with experts from the farm machinery manufacturing field, agricultural engineers and NLGI lubrication specialists participating. This article concludes the SPOKESMAN 1956 series on farm lubrication—our thanks to the following for their participation:

Panel Chairman M. L. Carter
Southwest Grease & Oil Corp.
Wichita, Kansas

Dale Hull
Iowa State College,
Ames, Iowa

Wendell Bowers
University of Illinois,
Urbana, Ill.

H. E. Achilles
Tide-Water Association,
Bayonne, N. J.

S. U. Franklin
Gulf Oil Corp.,
Pittsburgh, Pa.

N. A. Sauter
Deere & Company,
Moline, Ill.

T. E. DeVilliers
Cities Service Corp.,
New York, N. Y.

S. A. Bennett
Bennett Industries
Peotone, Ill.

F. B. Conron
British American Oil Co.
Toronto, Ontario, Canada

Owen Bandy
Lincoln Engineering Co.,
St. Louis, Mo.

Hal U. Fisher
California-Texas Oil Co.,
New York, N. Y.

E. W. Nelson
Continental Oil Co.
Ponca City, Okla.

J. W. Lane
Socony-Mobil Oil Co.
New York, N. Y.

C. J. Boner
Battenfeld Grease & Oil
Corp.,
Kansas City, Mo.

T. G. Roebner
Socony-Mobil Oil Co.,
Brooklyn, N. Y.

H. S. Elder
Illinois Farm Supply Co.
Chicago, Ill.

D. V. Ambler
British American Oil Co.
Toronto, Ontario, Canada

Question and Answer Session

Q. CHAIRMAN CARTER, Southwest Grease & Oil: I would like to have you elaborate a little more, Mr. Hull, on what you meant—what were the actual results that you found in the use of graphite grease in bearings?

A. HULL, Iowa State College: This is what the implement dealers reported to me in one area.

Q. CARTER: What was that?

A. HULL: Implement dealers have reported more than the expected number of failures in anti-friction bearings on trade-in combines. Normally they would expect to get "trade-ins" in pretty good mechanical condition. The dealer has to replace these bearings to recondition the machine. Now, any amount of money that an implement dealer has to put in to a used machine to dispose of it represents a loss to him. He has to discount the "trade-in" to cover the cost of bearing replacement.

Q. DE VILLIERS, Cities Service: I would like to ask Mr. Hull about the remark he made about farmers and farmer's activity being somewhat steeped in tradition. Would you say in your experience that it would be difficult to convert the farmers to another grease, than the one they have been using, if you had a better grease to furnish them?

A. HULL: No, not any more. Our experience with farmers—particularly in good farm practice, shows that they don't hesitate to accept a new practice or a new idea. They will take on new crop varieties rapidly. Our farm folks have a high level of intelligence. The majority of them are smart business men looking for methods to increase production efficiency and to cut costs. It isn't too hard to introduce a new idea, though it takes a little while for some of them. A good promotional sales story and good product education with your dealers should make introduction of a new grease product easier—particularly if you were to get a tricky little demonstration to show.

Q. FISHER, California-Texas Oil Co.: I would like Mr. Hull to expand a little bit, if he will please, on his remarks about the paper container that would lend itself most readily to the dispensing of greases for the farmer.



A. HULL: I wouldn't attempt to answer that. We know that farmers have accepted the one-quart and five-quart oil cans rather well. They like them because they can take that container to the field and the oil is always clean. I am not one to make predictions to really know how we are going to handle these farm greases in the future. Certainly the smaller package has some drawbacks such as increased retail price. There is a tendency too, with a smaller package, for a dealer to always take out the newer packages and leave the older ones because the lithograph on the can begins to look older and the farmer likes to have a new appearing package. I do know that at one time there was an organization marketing various types of greases in containers that fitted a dispenser and that made one gun do all the jobs. And there were storage and dispensing problems with that, too.

Q. ROEHNER, Socony-Mobil Oil Co.: Earlier this morning Hugh Hemmingway pointed out that lubrication has many ramifications, and in the case of lubrication of automotive bearings with grease, there is a safety factor. In other words, if someone doesn't get under that car now and then, they may not see a bearing about to fail and thereafter someone may have a serious accident. In the case of lubrication of farm machinery, which is getting more and more complicated, is there a safety angle similar to that in automotive lubrication? I am encouraged to think about that by the comment Mr. Bowers made of what he or his "grandfather done way back." They put their hand on the bearing to find out if it was warm, and that would be the red light and something would be done before the failure had occurred. Is there some sort of an angle, in other words, that could be mentioned along with just plain lubrication of bearings that would appeal to the farmer? And make sense to him?

CHAIRMAN: Mr. Bowers, would you like to answer the question?

A. BOWERS, University of Illinois: Well, you bring up safety in regard to the equipment and strange as it may seem, it's similar to safety of machinery. I think Mr. Hull would agree with me that it has taken several years to even make a dent on farmers on how to handle and use a corn picker. But Dale, I think we are making progress. And I think that we are now faced with the same future in equipment. We certainly put our own trust in lots of dif-

ferent equipment every day in automobiles. Take an automobile going down the highway at 70 miles an hour or more—if anything happens to that machine to take it off the road your life is gone. I don't think there is much question about it. However, the chances of that happening to an automobile without anything else around to cause it—its practically nil. I think when you get modern equipment developed, such as those with sealed bearings, I presume that is what you are referring to, where you simply have to put your trust in the equipment once it has been made to prove itself, then it becomes a matter of education on the part of the farmer.

Q. AMBLER, British American Oil Company, Canada: I would like to ask the panel members if they have had any luck in persuading the farmers, that if a grease isn't black it isn't good? Our experience seems to be that if it isn't dark, it isn't any darn good. I'd like to know if they have had any experience to refute that opinion?

A. BONER, Battenfeld Grease & Oil: I wonder if that doesn't follow the same experience as with motor oil. I recall a good many years back, motor oil was much darker and had a bright stock color. In other words the bright stocks at that time, and even the neutrals, had considerably more color. Some of the first solvent oil was very light and there was a sales resistance. Later people accepted the purity of the material. I think your modern man will accept a light colored lubricant after education, if not immediately.

Q. ACHILLES, Tide-Water: It seems to me from what the speakers have been talking about equipment, that it can go either of two ways. One: that we will have either a sealed-for-life bearing. Or Two: there will be centralized lubrication systems. Now do any of the people who are familiar with the equipment think possibly that the ultimate solution will be a combination of both and still manual handling of some of the more difficult bearing lubrication points?

A. SAUTER, Deere & Company: It's a little difficult to say. I think that when we look at the farm equipment industry, it is an industry that more or less develops by evolution, rather than by revolution. And I think the transition growth will be a very, very gradual one. I think over the



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past few years there has been quite a movement toward the sealed-for-life bearing and I would like to comment on what those words seem to mean since they mean various things to various people and there are several different approaches. One is the approach that one of the people from the universities mentioned, that in the Allis Chalmers sealed-for-life bearing on their disc harrow where the sealing is actually a sealed bearing within a compartment of grease. This is not what a lot of people think of when they think of a sealed bearing. They think more of a cartridge type bearing. I think to come back to your question that undoubtedly there is going to be a place where you can use the sealed-for-life bearings. There are going to be places where you can't and you will either go to the centralized system, or retain the present methods. Cost of course, in farm machinery, is very important right now. I got the impression from Mr. Bremier's talk that there was a lot of money in the farmer's pocket. Well, that may be, but right now the situation is not at all opportune as regards increasing the price of farm machines and that is what you would have to do to adapt more centralized lubrication to the equipment.

CHAIRMAN: Mr. Bandy do you have anything further to add to that on centralized lubrication?

A. BANDY, Lincoln Engineering: Well, there is only one question on the sealed-for-life bearing, in reference to the gentleman from John Deere on the cost of equipment. I wonder about the repair necessary by the dealer on replacement of the so-called sealed-for-life bearings. And we speak of rapid turnover of farm machinery. Well, if you trade an automobile in, it's a cinch that if you are going to get any trade in on it, it's because the automobile dealer can resell it. I think the same thing holds true with farm machinery. If you are going to trade it in, the farm machinery dealer has to resell the equipment some place and I am just wondering if there aren't a lot of cases where—the added cost of farm machinery actually is reflected in the fact that he has to replace these so-called sealed-for-life bearings.

A. SAUTER: I would like to touch on this as being a point that a farmer wants to trade-in his piece of machinery. We have the same situation going on in farm machines that we have in a lot of the industrial production machines today. It isn't too many years that go by before he can buy a

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machine that does two or three times what his machine will do, so that he sees the advantage of decreasing his unit cost and he trades his older machine in. Not because it is necessarily worn out but because he can buy a more productive machine.

CHAIRMAN: Another question?

Q. ROEBNER: I am bothered a little by this sealed-for-life type of bearing and I am glad to hear it defined. But isn't it true that at least to a certain extent, the use of so-called sealed-for-life bearings is almost an invitation to ignore those bearings because it is assumed that the designation "sealed-for-life" means operation forever? They don't ever say whose life that bearing is sealed for. Suddenly the bearing fails and the lives of other components of the machine are affected thereby. That is a point I was trying to touch on in my earlier question because isn't there such a thing as preventive maintenance, something that you do in order to guard against trouble happening down the road? And would sealed-for-life bearings, if not properly understood, lead to over-confidence and neglect?

CHAIRMAN: Thank you Ted. Who would you like to answer that? I believe that's Mr. Sauter's line.

A. SAUTER: Well, I think the point is well taken. I believe that it's the job of our engineering people, to engineer into these machines, the matter of life of the machine. It's well known statistically what the life of a farm machine is and we have people who go into quite considerable detail measuring the performance of the various components that go to make up the machine. I think before the bearing is adapted to the machine, you would pretty well know what the life of the bearing will only be. Now, obviously, if you have a 3600 rpm high inertia situation, such as you might have in a hammermill, you would give engineering consideration to the application. We have some of the high inertia applications such as that on other machines too, and by and large those bearings are set out with a grease fitting and no attempt is made to make those "sealed-for-life."

Q. NELSON, *Continental Oil Company*: Since this is a symposium on farm lubrication, I want to pass on a question that was asked of me recently by a farmer. This should probably be directed toward Mr. Bowers. Although the present trend is all toward bearing lubrication with pre-



packed bearings, centralized lubrication systems and pressure lubricated bearings, surely there still remains some machinery such as farm machines and garden equipment which are propelled by means of a chain drive. Which is the proper method of lubricating a chain? Should it be lubricated or non-lubricated? This particular individual wanted to know how he would get the best lubrication; should he use an oil, a gear lubricant, a grease, or no lubricant at all? He was of the opinion that if he didn't lubricate at all the chain would last much longer.

A. BOWERS: That is not a new question to me by any means and I don't think I've got any newer answer than I ever had. I believe the worst thing that a man can do is to put a brushfull of transmission grease on it every half day. I think the best thing he can do is to remove the chains before he starts a season or after he finishes and store them in light oil. Use SAE ten weight oil, and wipe the outside with a clean rag, put it on and forget about it.

CHAIRMAN: Another question?

Q. HOWARD S. ELDER, *Illinois Farm Supply Company*: Several of the speakers have mentioned that the life of the equipment would be vastly lengthened if it were properly lubricated. And of course the lubrication guide is always to be found in the operator's manual. But the problem on the farm is this—where is the operator's manual? Six months after the equipment is purchased—the manual is all too often misplaced or lost. I am wondering if the equipment companies could give some thought to some permanent form of instruction or lubricating guide on the equipment itself. The man that operates the equipment usually is the one who lubricates it and he doesn't have his lubrication guide as readily available as a service station operator. In other words, if there was some permanent lubrication guide for example, stamped on a metal plate on each piece of equipment itself, which would be readily available to whoever does the greasing, it might be a key for better lubrication, lubricated there on the farm.

CHAIRMAN: Mr. Sauter does your company have anything along this line?

A. SAUTER: Well, I might say that we are aware of the problem of the operator's manual not being available. We rely a good deal on our dealers to instruct the farmer in the proper care of the piece of equip-

ment and I hadn't had it put exactly to me the way that you did, in having individual recommendations for each machine. You are referring to the type of thing that is done on industrial machines. I can't say that it will be done, but it sounds like a reasonable thing.

Q. FRANKLIN, Gulf Oil Corporation: I notice that several of the speakers have mentioned multi-purpose type grease as being the most desirable in general. I wonder if that is correct? Are there any EP properties required around some farm machinery? It has been mentioned recently here by one of the men who asked a question about how to grease or lubricate a chain . . . an open chain. Is it so simple that a single multi-purpose grease can take care of all the points on the machinery? I wonder if Mr. Sauter, in setting his specifications for lubrication of various farm machinery thinks that a single multi-purpose grease takes care of all the situations?

A. SAUTER: If I can recollect my remarks correctly, I believe that we said the multi-purpose grease was not best to answer to every problem. I think the multi-purpose grease is a good answer to most problems and I think the problem in farm machinery today is not so much a requirement for EP but a requirement for sealing and I think that is the strong point of the multi-purpose grease. I want to ask Mr. Bandy a question. What is the maximum number of fittings that can be serviced, by what I call a one shot system, you call it something else.

A. BANDY: Well, it more or less depends upon quite a few factors and when you say one shot system, actually you cover quite a large range of centralized systems. You know there are quite a few on the market. It's possible to take care of as low as one bearing or as high as two thousand bearings. So it becomes a rather academic question as to how much money you want to spend.

CHAIRMAN: I would like to ask Mr. Bowers one question, which I have in mind here. I think perhaps he would be the best fitted. What do you think the farmer is most interested in when he goes out to lubricate his farm machinery? Is it his lubricating grease or is it the dispensing equipment that he uses to put a lubricant on the bearings?

A. BOWERS: I should have left with Hull. If you look at it from the standpoint of problems involved to himself, I think he



Question and Answer Session

would be more interested in the means of getting it to the bearing. I can't help but feel though, that the farmers put a lot of confidence in greases and oils. To a lot of farmers grease is grease, and oil is oil. And I think in a lot of cases even a film of oil in a slow speed or light load bearing is as good as a gun grease for no more than it has to do. I think a lot of farmers have the same viewpoint, that he would put more interest on getting it to the bearing than he would the material in it.

CHAIRMAN: In other words, he would leave the quality up to the manufacturer.

BOWERS: Yes, he would accept it, I believe.

Q. CONRON, British American Oil: I would like to ask Charlie Boner if he would care to technically define an "all purpose grease" for farm use.

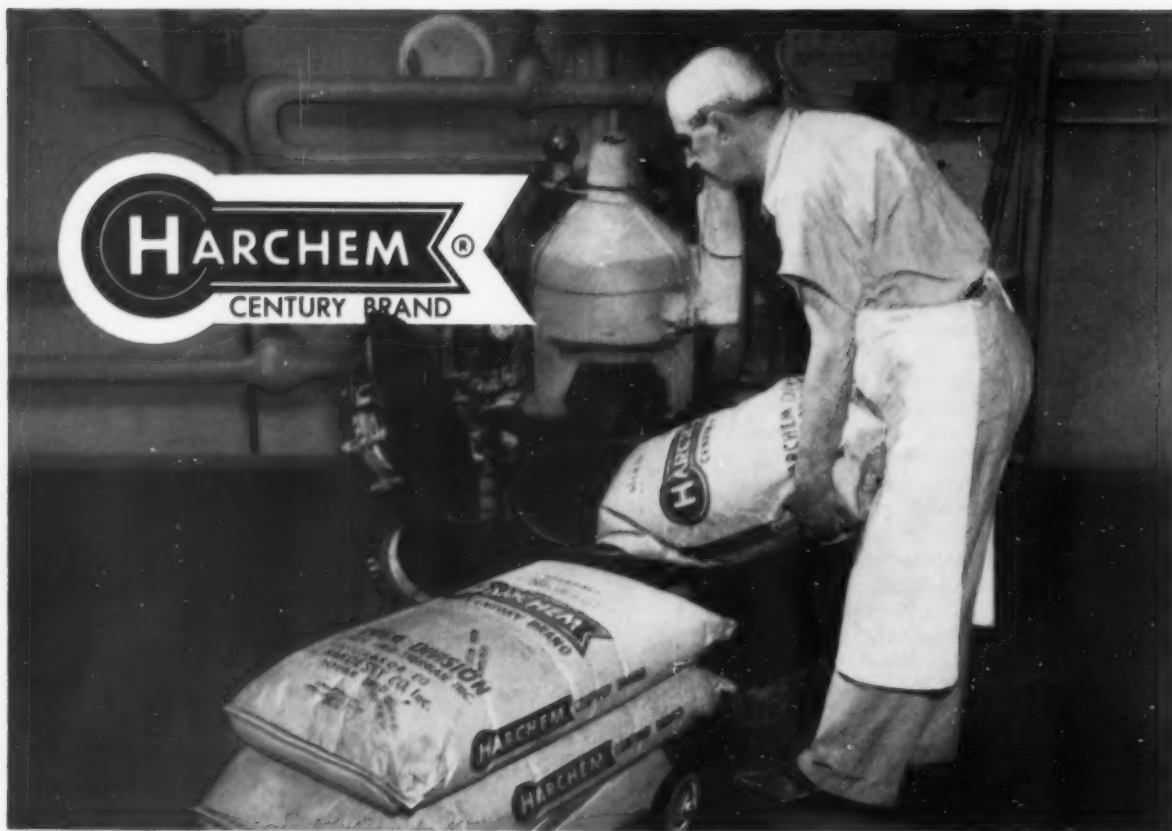
A. BONER: Don't you think perhaps the term "all-purpose" is wrong? We talk about multi-purpose, instead of the term you use. I think if you will take the characteristics of average products that were marketed in the past—water pump, wheel bearing, chassis lube product and combine them, that you will have a multi-purpose product. I don't think that even this will fill every job.

CONRON: I'm still not satisfied. I believe that's saying "I'm a babe in the woods" in this matter about the grade, any NLGI grade? Would you even care to hazard an opinion about the metal used as the base of the all purpose? In other words, would you say it was a number two NLGI lithium grease—would that be a multi-purpose grease that would do all these jobs?

BONER: Unfortunately, there have been a great variety of lithium based greases made and I should point out that there have been some very, very ordinary lithium greases marketed. And there probably will continue to be.

A. LANE: In reply to the question our Canadian visitor expressed, I would like to point out that multi-purpose greases are marketed in several NLGI grades—at least two. That would indicate in itself, that there is no such thing even as an "all purpose" multi-purpose grease.

CHAIRMAN: I appreciate the attention you gentlemen have given this discussion this afternoon and so I turn this program back to Mr. Lane.



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Patents and Developments

Magnesium-Lithium-Sodium Grease

Magnesium base greases are well known in the art of lubricating grease manufacture. They are characterized by excellent appearance and by their resistance to shear breakdown. It has been found, however, that magnesium base greases are somewhat lacking in stability properties at sustained high temperatures. When utilized for the lubrication of moving metal parts at high temperatures for long periods of time these otherwise excellent lubricants have been found to harden to an undesirable extent and to allow oil to separate. This combination of a tendency to harden and to separate oil is an undesirable one and many attempts have been made to modify magnesium base greases to remove it.

One line of research in this direction has been the incorporation of metallic soaps, such as sodium soaps, potassium soaps and the like. This has not been satisfactory in that while the addition of these soaps decreases the tendency of the magnesium grease to harden, it increases the amount of oil separation under conditions of sustained high temperatures.

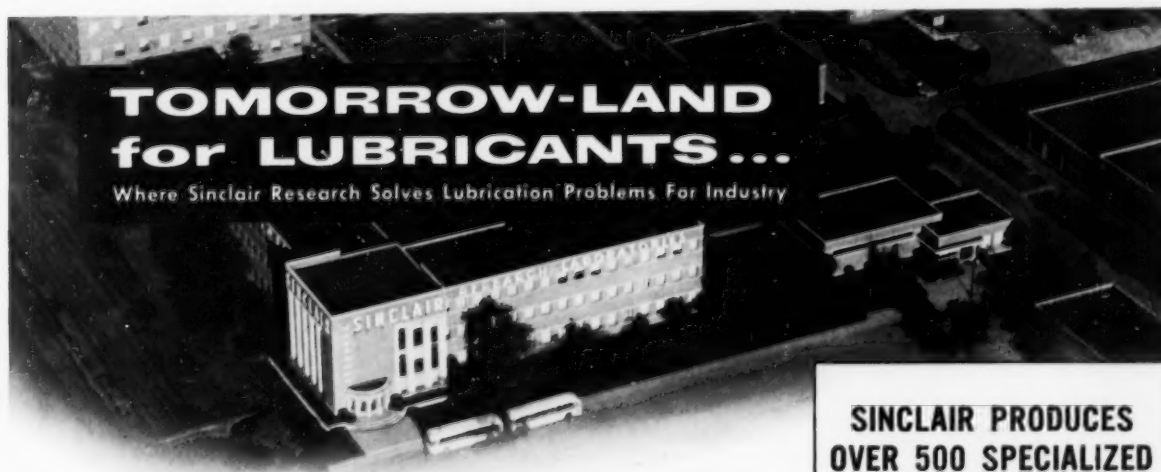
According to U. S. Patent 2,733,209 issued to Esso Research and Engineering Company, the incorporation of minor amounts of both a sodium soap and a lithium soap in a magnesium base grease results in a composition that has the excellent structure stability and appearance of the magnesium greases of the prior art and in addition removes their tendency to become hard and to separate

oil under conditions of sustained high temperatures. This combination of properties makes these new grease compositions extremely useful in lubrication applications such as in the sealed bearings of electric motors, railroad anti-friction bearings, and automotive bearings. The compositions demonstrate a combination of desirable properties which are surprising and unexpected when compared with the results of incorporating either sodium or lithium soaps alone in a magnesium base grease.

The greases are prepared by thickening to a grease consistency a lubricating oil base stock with a mixture of the magnesium, lithium and sodium soaps of any of the well known fatty acids familiar to lubricating grease makers. Care must be taken only to prepare the most insoluble soap first, followed by the lesser soluble and most soluble soaps in the order.

Briefly, the preparation involves the thorough admixture of the fatty acids used in a portion of the mineral oil base and the application of heat until a homogeneous mixture is obtained. A slurry or solution of the metallic base usually in the form of hydroxide is then added and the mixture heated to dehydration temperatures. On completion of dehydration the total mixture is heated to about 300° F., the balance of the mineral oil added and the final product cooled without stirring.

The thickening agent of the greases of the invention consists of a mixture of the magnesium, lithium and sodium soaps of the above mentioned acids. The ratio of



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the three soaps used will vary from about 5 to 1 to 1 mols of magnesium, lithium and sodium soaps, respectively, to about 1 to 5 to 5 mols. The preferred compositions of the invention will contain about one-half molar proportion of lithium and sodium soaps to each mol of magnesium soap.

The amount of the total soap mixture that is utilized in preparing the greases will depend upon the consistency desired in the final product. Ordinarily from about 5.0% by weight to about 30.0% will be used with from 10.0% to 25.0% by weight of the total mixture being especially preferred.

The following example is given as to the preparation of a typical grease composition:

Ingredients:	Formulation	Weight Per cent
Hydrogenated fish oil acids.....		22.00
Magnesium hydroxide		4.15
Lithium hydroxide		0.70
Sodium hydroxide		1.00
Mineral lubricating oil (70 SUS/21 0° F.).....		72.15

Preparation

The total charge of the hydrogenated fish oil acids and one-third of the mineral oil were mixed and heated until a homogeneous solution was obtained. An oil slurry of the magnesium hydroxide was then added and the magnesium soap allowed to form. Thereupon an aqueous solution of lithium hydroxide was added and the lithium soap formed. Upon completion of this reaction an aqueous solution of the sodium hydroxide was added and the

sodium soap was prepared. The total mixture was then heated to 240° F. and dehydrated. After all the water was driven off the temperature was raised to 300°F. and the remainder of the mineral oil was added gradually with stirring. Upon completion of the addition of the mineral oil the product was cooled without further stirring.

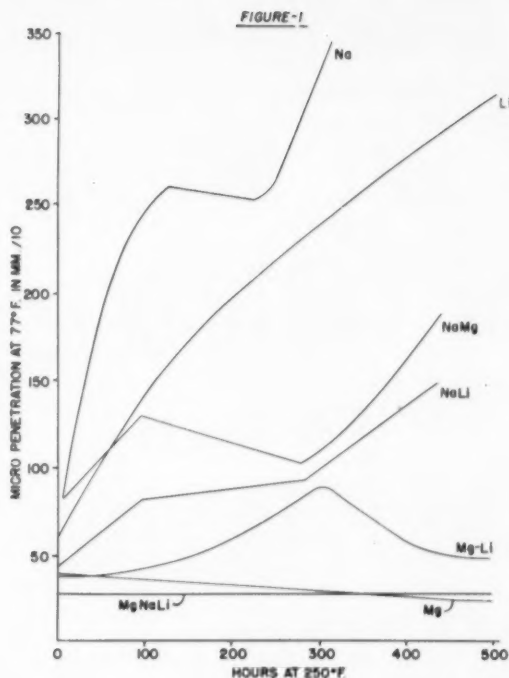
Test results in various greases are depicted in Figure 1. The micropenetration of the samples are plotted against the number of hours subjected to the test temperature. It will be observed that whereas the patented composition (Mg-Na-Li) remained uniformly stable for the duration of the test, the greases of the comparative examples softened to a great extent with subjection to the test temperature.

Greases Containing an Orthosilicate

Ester oils have been used as lubricating media in oil compositions and greases.

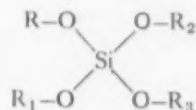
Such aliphatic ester oils are liquid saturated aliphatic normal esters of saturated aliphatic carboxylic acids, which esters are further defined by the fact that their boiling point is 150° C. or higher. This class of liquid aliphatic esters has been recognized by the art as a source of synthetic lubricating oils and some of these esters have been used with metal soaps for making greases. The art is also aware that metal soaps accelerate the thermal oxidation of these liquid aliphatic esters and has added antioxidants to the greases to improve their oxidation stability.

In U. S. patent 2,735,816 issued to Robert L. Merker and William A. Zisman of Washington, D. C., there is



disclosed a new class of antioxidants for improving the oxidation stability of the aliphatic ester oils which contain metal soaps, especially at elevated temperatures.

It has been found that the oxidation stability at elevated temperatures of aliphatic ester oils which contain metal soaps can be improved by the addition thereto of small amounts of an orthosilicate which is aromatic or hydroaromatic or mixed aromatic-hydroaromatic in character. The orthosilicate antioxidants contain an aromatic or hydroaromatic group on each of at least two of the ester oxygen atoms of the molecule, with a lower alkyl group being attached to such of the ester oxygen atoms as are not satisfied by an aromatic or hydroaromatic group. These orthosilicates have the general formula:



wherein R, R₁, R₂ and R₃ each represent an aromatic or a hydroaromatic radical, or in part represent a lower alkyl radical of up to 6 carbon atoms where only two or three of the ester oxygen atoms of the orthosilicate molecule are attached to an aromatic or hydroaromatic radical. The radicals R, R₁, R₂ and R₃ may be the same or different aromatic and hydroaromatic groups and the same or different lower alkyl groups where two such are present in the orthosilicate molecule. The radicals R, R₁, R₂ and R₃ may be aryl or hydroaryl groups which may

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be substituted or not by groups such as, for example, alkyl, cycloalkyl, aralkyl, alkoxy, aryloxy, hydroxy, carboxy, carbalkoxy, nitro, amino, alkylamino, arylamino, etc., groups.

A preferred class of the orthosilicate compounds as antioxidants for the new fluid compositions are those in which the aromatic and hydroaromatic groups attached to the ester oxygen atoms of the orthosilicate molecule are mono- or dinuclear and more, preferably mono- and dinuclear aryl and hydroaryl groups which may or may not contain lower alkyl groups, for example, methyl, ethyl, isopropyl, butyl, tertiary butyl, hexyl, etc., groups. Within this preferred group of orthosilicate compounds is to be mentioned in particular, tetraphenyl orthosilicate and tetracyclohexyl orthosilicate.

The stability to oxidation, especially at elevated temperatures, e.g. at 80 to 150°C., of the aliphatic ester oil-metal soap compositions can be enhanced by the addition thereto of one of the antioxidant orthosilicates in concentrations within the range of about 0.1 and 10% by weight therein, with a preferred general range for the addition of the antioxidant being from about 0.1 to 5% by weight. The amount of the antioxidant added will depend upon the activity of the individual orthosilicates and the particular aliphatic ester oil-metal soap composition. The orthosilicate compound is dissolved or dispersed in the aliphatic ester oil depending upon its solubility therein.

The invention is illustrated in greater detail by the following example in which the orthosilicate compound

was incorporated in a lithium soap-diester grease and subjected to an accelerated oxidation test to determine its efficacy as an antioxidant for the metal soap-diester oil composition at elevated temperatures. The greases were prepared in conventional manner by heating the aliphatic ester oil and metal soap together under stirring to form a clear solution, quickly cooling the solution to form a gel and working the gel through a screen to obtain a grease of uniform consistency (about 10 workings through a Hain Microworker). The antioxidant was mixed into the grease and the whole then worked through the screen as before to obtain uniformity of admixture. The control grease or blank was made up to contain the same relative proportion of the metal soap and diester oil in each instance but without the presence of the orthosilicate anti-oxidant. The accelerated oxidation tests were carried out in accordance with Navy Specification AN-G-3a (2) using a Norma-Hoffman bomb apparatus.

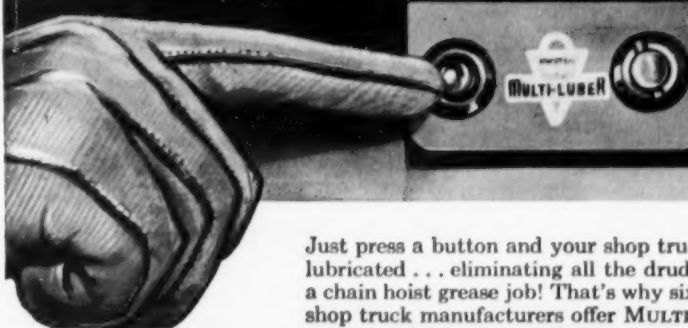
Example

A grease was prepared to contain 16.2 mol per cent (11.12% by weight) lithium stearate, 80.8 mol per cent (87.68% by weight) di-2-ethylhexyl sebacate and 3.0 mol per cent (1.2% by weight) tetraphenyl orthosilicate. A control grease was made up in the same manner to contain only lithium stearate and di-2-ethylhexyl sebacate in the same relative proportions.

Twenty gram samples of each of the two greases were placed in separate Norma-Hoffman bombs which were then sealed, heated to 100° C. and oxygen presses thereinto at 110 pounds per square inch. The oxidation test

Continued on page 60

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Southwest Grease Appoints New Salesmen



R. E. Burns



J. H. Diefenderfer

John H. Diefenderfer has joined Southwest Grease & Oil Corp. after an extensive orientation in his territory, which will include Washington, Oregon, California, Nevada, Arizona, Utah, Idaho, Montana and parts of Wyoming and Colorado. Mr. Diefenderfer will reside in Los Angeles, California.

Ross E. Burns, Jr. has accepted the responsibility of Southwest's Northeast territory under the direct supervision of Wilson Simmons. Mr. Ross will travel North Dakota, South Dakota, Nebraska, Minnesota, Iowa, Wisconsin, Illinois, Indiana and parts of Missouri. He is presently living in Joplin, Missouri.

Battenfeld's Daniel Host to Belgium Member

William Claessens, Director of Usines Purlina-Palmarine in Ertvelde-Rieme, Belgium recently visited Kansas City, Missouri. A. J. Daniel, President of Battenfeld Grease & Oil Corp. and Treasurer of National Lubricating Grease Institute acted as NLGI host and representative. Mr. Claessens is responsible for the manufacture of lubricating greases for his firm which also manufactures aluminum stearate and fatty acids.

Kerr-McGee Elects L. A. Woodward Vice President

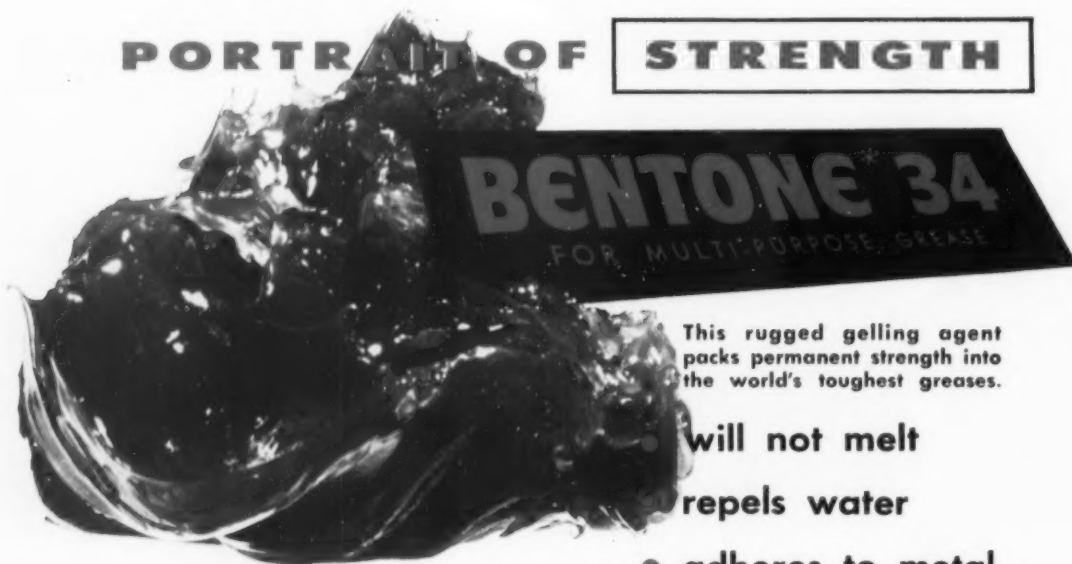
Mr. D. A. McGee, president of Kerr-McGee Oil Industries, Inc., has announced the election of L. A. Woodward as administrative vice president of that company. Woodward was formerly president of Mid-West Refineries, Inc. of Grand Rapids, Michigan.

Promotion of Two Girdler Engineers Announced

John E. Slaughter, Jr., vice president of The Girdler Company, Louisville, Ky., has announced two promotions in the Votator Division, which he heads.

Robert E. Deatz has been moved up to assistant chief engineer in charge of project and design engineering, which is under the direction of Bruce Miller. He was formerly chief project engineer of the Division's fats and oils section.

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Mr. Deatz obtained his B. S. degree in chemical engineering at Kansas State College. He joined The Texas Company as a process engineer in 1942 and became a member of Girdler's technical staff in 1947.

Louis P. Bornwasser has succeeded Mr. Deatz as chief project engineer of the fats and oils section. He obtained his B. Ch. E. degree from Virginia University. Before joining Girdler in 1948 he was associated with E. I. Du Pont de Nemours Company and the Monsanto Chemical Company.

Both Mr. Deatz and Mr. Bornwasser are members of the American Institute of Chemical Engineers, American Chemical Society and the American Oil Chemists Society.

Girdler is a division of the National Cylinder Gas Company, Chicago.

Girdler's Votator Division designs and manufactures processing apparatus and designs and erects complete processing plants for the fatty oil, food and chemical industries.

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SHELL ANNOUNCES MANUFACTURING AND MARKETING PERSONNEL CHANGES



M. P. Love

F. S. Clulow

M. P. Love Manufacturing Vice President

Shell Oil Company has announced the appointment of M. P. L. Love as manufacturing vice president. He will succeed F. S. Clulow who will retire about the middle of this year after nearly 36 years of Shell service.

Mr. Love joined Shell in 1935 as a junior chemist at the Wood River, Ill., refinery. Two years later he became a senior research chemist. After assuming positions of increasing responsibility at Wood River, in New York and in the Houston Research Laboratory, he became manager of the manufacturing research department in New York in 1948.

In 1951, he became manager of the Houston refinery, where he remained until August, 1954. Recently he has been on a special assignment.

Mr. Love expects to assume his new post in the middle of this year.

Clulow Retires

Mr. Clulow was named a department manager at the Martinez, Calif., refinery in 1930 following ten years of service with the Shell organization.

In 1934 he became manager of the East Chicago, Ind., refinery and in 1938 returned to the West Coast to become manufacturing vice president with headquarters in San Francisco.

In 1949, following the consolidation of the Pacific Coast and East of the Rockies manufacturing organizations, Mr. Clulow came to New York to become manufacturing vice president for all Shell refineries.

Five Marketing Advancements

Five personnel changes in the marketing department of Shell Oil Company were announced by J. G. Jordan, vice president. The changes are effective in June.

M. Bogstahl, operations manager of the St. Louis division, has been named manager of the marketing engineering department, head office, New York. He succeeds C. J. Nobmann, who retired after thirty-three years' service.

J. S. Hoppock, Detroit sales manager, will become operations manager, St. Louis.

E. F. McGee, operations manager, Albany division, will become sales manager, Detroit.

H. Wearne, acting manager of the marketing engineering department in New York, will become operations manager, Albany.

Houghton & Co. Appoints R. K. Smith

Dr. Robert K. Smith has been appointed Manager of Research for E. F. Houghton & Co., 3rd and Lehigh Ave., Philadelphia, Pa., manufacturers of industrial oils, packings, metal working products and chemicals.

Dr. Smith, 36, was formerly asso-

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The new unsaturated alcohols are liquids—almost water-white. The principal component of UNADOL 40 is *linoleyl* which possesses two double bonds. The principal component of UNADOL 90 is *linolenyl* which contains three double bonds.

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ciated with the Houdry Process Corporation, Marcus Hook, Pa., as Chief of the Exploratory Research Section.

He is a graduate of Pennsylvania State College with Bachelor and Master degrees in Organic Chemistry, and received the degree of Doctor of Philosophy in Physical - Organic Chemistry from Princeton University. Before joining Houdry he was associated with the Kendall Refining Company and previously with the Standard Oil Development Co. in a research capacity.

Du Pont Names New Managers

R. F. Harwick



A new regional manager, two new additive managers, a new district manager, and a new account manager have been named for the Du Pont Company's Petroleum Chemicals Division, it was announced by David H. Conklin, director of sales.

Robert F. Harwick, formerly assistant manager of the Akron district of the Elastomers Division, sales promotion manager of the Petroleum Chemicals Division, and Gulf Coast assistant regional manager of that division, has been named manager of the Western region. George L. Tyler becomes assistant manager, and William B. Gest has been named manager of the newly created Cleveland, Ohio, district.

Edison D. Jeffus, who has been manager of the Eastern Regional Laboratory, has been named to the newly created post of additives manager for the Gulf Coast region, headquartered in Houston. William W. Wingate has been appointed to the other new position of additives manager for the Mid-Continent region with the main office in Tulsa. Richard O. Braendle moves to assistant to the manager of additives sales in Wilmington, the post formerly held by Mr. Wingate.

William K. Park has been transferred from the Central region to the Eastern region as an account manager with headquarters in New York City.

NLGI SPOKESMAN

Emery's Research Requires Two New Sales Groups

Sparked by the multi-million dollar expansion in research and plant facilities of Emery Industries, Inc., K. K. Boyd, vice president in charge of sales and purchases, announces the formation of two new sales departments to replace the present single chemical sales operation.

This new line-up will provide better and more expert technical help to the many industries served, Boyd explains. It will bring related product lines into closer alignment and make possible even better technical sales



G. W. Boyd

R. F. Brown

attention for new products and old products alike.

Robert F. Brown, for nine years the well-known sales manager of Emery's present Chemical Sales Department, will head one of the new sections, with Robert Hull and Vern Colby as assistant sales managers. This new group, called the Organic Chemical Sales Department, will handle the sales of all non-fatty Emery products. Included are azelaic and pelargonic acids along with plasticizers, esters and diesters including those designed for special lubricants, polymerized (dimer) acids, textile processing oils and Twitchell emulsifying bases.

G. William Boyd, for the last nine years sales manager of Emery's rapidly growing Sanitone Division, becomes sales manager of the new Fatty Acid Sales Department. In addition to Emery's standard fatty products and stearic and oleic, all animal and vegetable fatty acids, hydrogenated fatty acids and glycerides, and derivatives of castor oil will be handled by this group.

According to A. W. Schubert, Emery's executive vice president, this move is an important one in Emery's long range planning to develop better coverage of the industries now being

served. With the anticipated growth from both new products and expanded utility of present products,

these new sales departments will be expanded as necessary to follow through on these assignments.



L. N. Hodson

Lee N. Hodson, Sr.—Deceased

Mr. Lee N. Hodson, Sr., President and General Manager of The Hodson Corporation passed away suddenly at the Roseland Community Hospital, Thursday, March 22, 1956.

Mr. Hodson was born in the Hamilton Park area on the south side of Chicago on November 5, 1892. He lived most of his life in the Michigan and Chicago area. In his lifetime he represented various leading manufacturers.

Mr. Hodson leaves his wife, two sons, two brothers, and a sister.

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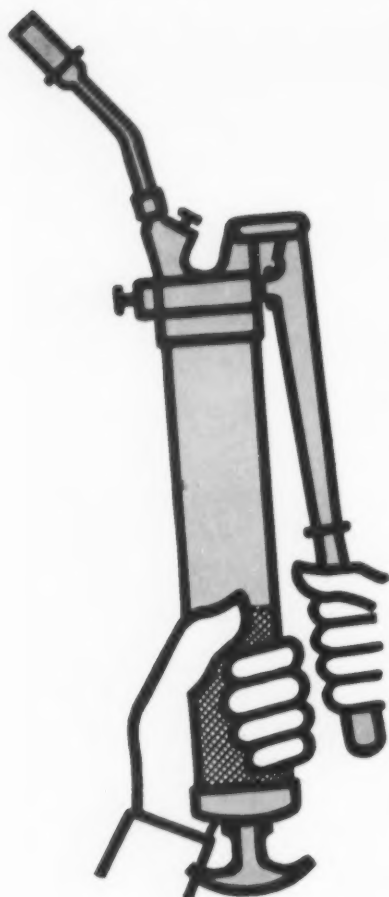
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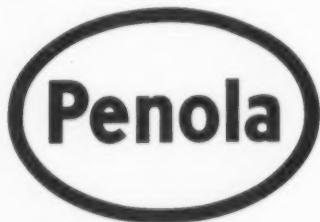


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Industry NEWS

Lincoln Engineering Merges With McNeil Company

Effective April 27, 1956 the Lincoln Engineering Company of St. Louis, designer and manufacturer of lubricating equipment, merged with The McNeil Machine & Engineering Company, Akron, Ohio, by vote of the shareholders of both organizations. McNeil is the surviving corporation and the new name of Lincoln is Lincoln Engineering Company, Division of The McNeil Machine & Engineering Company.

Mr. Alex P. Fox, former president of Lincoln, and Mr. Foster Holmes, former Executive Vice President, were elected to the McNeil Board of Directors headed by Mr. Charles F. Safreed, President and one of the original founders of the McNeil Corporation. The McNeil Board elected Mr. Fox Vice President of the Lincoln Division. Mr. Holmes was elected Assistant Treasurer and Mr. Gorman A. Wolf, former Secretary of Lincoln, was elected Assistant Secretary, to serve the Lincoln Division.

Mr. Jonathan Kludt, former Vice President In Charge of Production of Lincoln Engineering Company, was elected to serve as General Manager of the Lincoln Division; Mr. John E. Renner, former Vice President in Charge of Sales will continue to head the sales department as General Sales Manager; and Mr. Carl H. Mueller former Vice President of Engineering will remain in charge of this department as Director of Engineering. An Operating Committee was in turn appointed, consisting of: Mr. Fox, Mr. Holmes, Mr. Kludt, Mr. Mueller and Mr. Renner.

In addition to the merger with Lincoln Engineering Company, the McNeil corporation previously acquired two other companies, the Cleveland Crane and Engineering Company of Wickliffe, Ohio, manufacturer of overhead material handling equipment, shears and presses; and the Hamlin Metals Products Corporation, also of Akron.

The McNeil Machine & Engineering Company manufacturers presses for molding and curing rubber tires, and is a supplier of this equipment to all well known rubber companies.

Alpha Molykote Issues "Lubrication Newsletter"

"Lubrication Newsletter," the first issue of a technical house organ issued by The Alpha Molykote Corporation, Stamford, Connecticut, carries as its lead story a discussion of press fitting with molybdenum disulfide lubrication. The article deals with press fitting of plain bearings, ball and roller bearings, gears, sprockets, clutches, flywheels, etc. and includes a thorough delineation of the mathematics of press fits. Curves show press and shrink fit variables as well as elastic changes in diameters of press fits.

Also included in "Lubrication Newsletter" is an excerpt of the proceedings of the First International Congress on Molybdenum Disulfide For Lubrication as reported by the German Mineral Oil Association.

The Alpha Molykote Corporation will add to its mailing list, the names of technical persons who can make use of this lubrication information.

WANTED:

GREASE CHEMIST

Chemist or Chemical Engineer for Development and Technical Service work on Lubricating Greases. Excellent opportunity. New York City area. Give full particulars of past training and experience. Reply to

NLGI SPOKESMAN
Box No. J-2

NLGI SPOKESMAN

Gross Co. Announces New Oleic Acid

A new Oleic Acid which is a low Linoleic White Oleine was introduced this month by A. Gross & Company, 295 Madison Avenue, New York 17, N. Y., manufacturers of stearic acid, red oil, glycerine and fatty acids, according to Eugene W. Adams, Vice President.

Marketed under the Gross trade name "GROCO 5L," the new product is a premium Oleic Acid of extremely light color and low Linoleic Acid content. This gives it extraordinary stability and resistance to oxidation.

According to Mr. Adams, recent production has resulted in Oleic Acids with a Linoleic content well under the established maximum of 3.5%. The oil has a low titre under 5°C. and an Iodine Value of 86 max.

GROCO 5L has the following specifications:

GROCO 5L

Low Linoleic White Oleine

Titre	5°C. max.
Color 5¼" Lovibond Red ...	1 max.
Color 5¼" Lovibond Yellow...	10 max.
Color Gardner 1933	2 max.
Unsaponifiable	1% max.
Saponification Value	200-205
Acid Value	199-204
Iodine Value	86 max.

Samples of GROCO 5L are available upon request.

Kerr-McGee Absorbs Deep Rock as Division of Company

Effective May 1, Kerr-McGee Oil Industries, Inc. will absorb its marketing subsidiary, Deep Rock Oil company, as a division of the company.

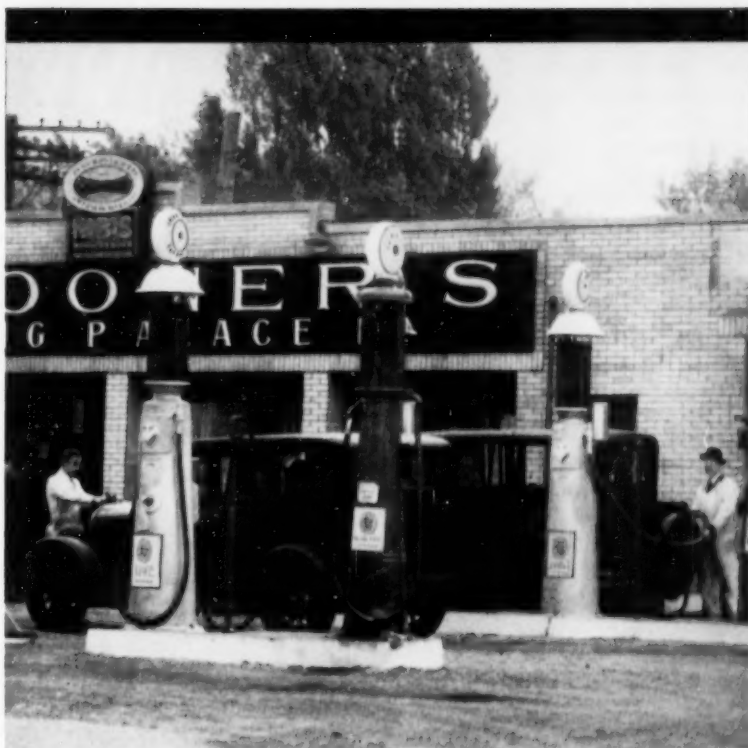
Since purchasing Deep Rock in April, 1955, Kerr-McGee has been marketing products through more than 800 service stations in 23 states.

Kerr-McGee will continue to use the 41-year-old Deep Rock brand name and the familiar yellow and blue rectangle on service stations and products.

Back of the new Deep Rock division of Kerr-McGee lies a history that began with discovery of the Cushing, Oklahoma, oilfield on April 1, 1912.

Early-day company officials had recognized the need for a trade name under which to market casinghead gasoline produced on their leases.

JUNE, 1956



Since "way back when," Monsanto has been helping processors like you with the chemical problems of petroleum modifiers

Modern Example: CONTROLLING THE CORROSIVENESS OF LIGHT PETROLEUM FRACTIONS

The problem, of course, was to find an inhibitor—one that in very, very low concentrations would keep light distillates from chewing up metal. It took some searching—but Monsanto chemists came up with Santolene C; just .0015% of it in the stock cut scale formation in a cargo tanker by 2 tons per month.

When you want a base stock modified to meet laboratory specifications before starting expensive field testing... send the base stock and your specifications to Monsanto. Monsanto can supply you with Chemical Additives for almost any job an additive can do: Corrosion inhibitors • Detergents • Viscosity index improvers Gear lubricant fortifiers • Fuel oil clarifiers • "Oiliness" improvers.

For more information,
write to:

Organic Chemicals
Division
Monsanto Chemical
Company
Department OA-1
St. Louis 1, Missouri



Santolene Reg. U. S. Pat. Off.

They based their search for a name on the term "rock oil," an early-day term for petroleum. In searching for a companion word, they settled on the word "deep" as connoting depth of richness and quality.

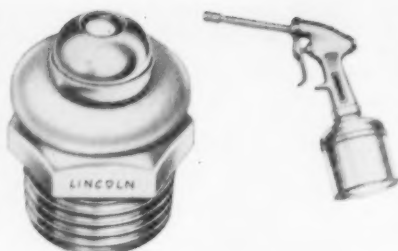
Since last summer Deep Rock refining and marketing activities have been directed from Kerr-McGee's Oklahoma City headquarters.

Dean A. McGee, president of Kerr-McGee, said Deep Rock's new status as a marketing division is "a planned step in the integration and expansion of the company."

Lincoln Announces New High Pressure Oiler

The first hand-operated high-pressure oil gun for contacting standard lubrication fittings has been announced by Lincoln Engineering Co. It is now possible to have positive pressure lubrication with oils—as well as flushing and cleansing of bearings which require periodic applications of fluid lubricant.

Used with the pressure oil gun, are



metal rings anodized in various colors. They are slipped over the head of each fitting to provide a color coding system for all bearings requiring oil—thus simplifying standardization. In addition, these rings make the accidental use of a grease gun impossible.

Address inquiries to Lincoln Engineering Company, 5702-30 Natural Bridge Avenue, St. Louis, 20, Missouri.

Swift Announces New Vegetable Fatty Acid

A new hydrogenated fatty acid of vegetable origin has been developed for lubricating greases by Swift & Company, Industrial Oil Dept., Hammond, Indiana.

Identified as Swift's Hydrogenated Fatty Acid No. 62 the new product has a high melting point and high stearic acid content, the company reports. The following specifications are offered for the product:

Titer	61-64°C
Iodine Value	3 Max
Acid Value	194-204
Saponification Value	195-205
Lovibond Color (5 1/4" Col.)	15Y/1.5R Max

The new fatty acid is shipped in 50 pound net weight paper bags. For further information or sample quantities contact Swift & Company, Industrial Oil Dept., 1800-165th Street, Hammond, Indiana.

Lithium Corp. Produces Lithium Dispersions

Lithium Corporation of America, Inc., Minneapolis, Minnesota, has announced the successful production of dispersions of lithium metal. Interest in the possibilities of these dispersions has been heightened by recent reports on their use as catalysts in the production of the new "natural" rubber.

The company's Product Research & Development Department is making available instruction sheets de-

scribing the methods for laboratory preparation of these lithium metal dispersions in such dispersing mediums as mineral oil, petrolatum and wax. Particle sizes in the range of 5 to 20 microns are possible by use of these techniques. It is expected that some time in the near future prepared dispersions in selected media will be available for sale through the company's Sales Department.

Lithium Corporation Announces 4th Supplement to Bibliography

Lithium Corporation of America, Inc. has announced publication of Supplement No. 4 of the Annotated Bibliography On The Use Of Organolithium Compounds In Organic Synthesis. Distributed without charge to those engaged in chemical research, the Original Bibliography and its Supplements are offered as a reference work and guide to research on Lithium and its compounds.

Mallinckrodt and Climax Await Joint Operation Okay

An agreement has been signed between Climax Molybdenum Company and Mallinckrodt Chemical Works to submit to the Atomic Energy Commission a joint proposal for the construction and operation of a privately owned plant for the refinement of uranium compounds, it was announced by Arthur H. Bunker, president of Climax, and Charlton MacVeagh, vice chairman and treasurer of Mallinckrodt.

The agreement further states that in the event the proposal is accepted a jointly owned company would be formed to construct and operate such facilities. Under terms of the agreement Climax would be majority owner of the resultant company.

DARLING'S

FATTY ACIDS

ESPECIALLY FOR

GREASE MAKERS

STEARIC ACID

OLEIC ACID

RED OIL

HYDROGENATED
FATTY ACIDS

HYDROGENATED
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People that count"

THE SAVINGS WITH INLUCITE 21

Lithium-base Multi-Purpose Grease

**REALLY
ADD
UP!**



- ▲ Service stations and fleet owners are finding that **INLUCITE 21**, the amazing "one grease-one gun" lubricant, saves time, saves labor, saves product cost, saves inventory and it's superior to every specialized grease it replaces!
- ▲ Profits jump for service stations two ways — through savings in man hours, inventory and product cost, and through increasing the number of grease jobs done.
- ▲ Savings multiply for fleet owners too . . . fewer greasings, less downtime, greater protection under all operating conditions.
- ▲ **INLUCITE 21** is the registered trade name of the original lithium-base grease made under our exclusive patents.

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CORPORATION**

New Orleans, Louisiana

Manufacturers of Quality Lubricants

AVIATION • INDUSTRIAL • AUTOMOTIVE • MARINE

With Research Comes Quality ★ With Quality Comes Leadership

Patents & Developments

Continued from page 51

was run for a period of 300 hours during which time the temperature in bombs was maintained at $100^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$.

Bomb pressure readings were made at intervals over the 300 hour period, which readings were accurate to ± 1.0 p.s.i. Consumption of oxygen by the grease and thereby oxidation of the same is indicated by drop in the oxygen pressure in the bomb. Readings of the pressure in the bomb containing the control grease showed a rather sharp drop even in the earlier stages of the run (up to 50 hours) which was progressive until at the end of the 300 hours, the pressure stood at 50 pounds per square inch, indicating a substantial amount of oxidation to have taken place in the unprotected grease. On the other hand, pressure readings in the bomb containing the grease to which the tetraphenyl orthosilicate had been added showed no drop in pressure over the 300 hour period, indicating the orthosilicate compound to be a particularly effective antioxidant for the grease.

The aliphatic ester oil-metal soap compositions may be blended with miscible proportions of other lubricating

oils, such as petroleum oils and polyoxyalkylene oxides having one or both terminals ending in an alkyl ether group, e.g., the butoxy group. Antioxidants of types other than the orthosilicates may be used in the compositions in conjunction with the tetra-aromatic and tetrahydroaromatic orthosilicate antioxidants, such as phenolic and aromatic amine type antioxidants which are active as antioxidants at the lower temperatures, e.g. below 100°C ., and thereby will impart supplemental protection to the aliphatic ester oil-metal soap compositions at temperatures occurring during storage or during other low temperature nonoperating periods, e.g., cyclohexyl phenol, p- hydroxy diphenyl, 4-t-butyl-2-phenylphenol, thymol, catechol, phenyl-a-naphthylamine, etc. Other antioxidant compounds of the non-silicate type are dialkyl selenides, e.g., dialuryl selenide, and phenothiazine. Some of these supplemental antioxidant compounds are active at the elevated temperatures also, for example, dilauryl selenide and phenothiazine.

FUTURE MEETINGS of the Industry

JUNE, 1956

Continued on page 62

- 10-15 API Division of Production (midyear standardization conference), Brown Palace and Cosmopolitan Hotels, Denver.
- 11-15 Oil Heat Institute (34th annual convention), Park Sheraton Hotel, New York City.
- 11-15 Oil Heat Institute (21st national oil heat and air conditioning exposition), Coliseum, New York City.
- 12-13 Petroleum Packaging Committee, Statler Hotel, Boston, Massachusetts.
- 13 Pennsylvania Grade Crude Oil Association, Pennhills Club, Bradford, Pa.
- 14-16 The American Society of Mechanical Engineers (applied mechanics conference), University of Illinois, Urbana, Ill.
- 17-20 American Society of Agricultural Engineers (49th annual meeting), Roanoke Hotel, Roanoke, Va.
- 17-21 The American Society of Mechanical Engineers (seminar meeting), Statler Hotel, Cleveland.
- 17-22 ASTM 59th Annual Meeting and 12th Apparatus Exhibit, Chalfonte-Haddon Hall, Atlantic City, N. J.

**SIGN OF
CORRECT
LUBRICATION**



Makers and Marketers of

**Mobil
Automotive**
Oils and Greases

Industrial
Oils and Greases

SOCONY MOBIL OIL CO., INC., and Affiliates
MAGNOLIA PETROLEUM COMPANY
GENERAL PETROLEUM CORPORATION

**McGEAN 30% LEAD
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Consistently uniform in metallic content and viscosity

Fully clarified by filtration

Non-Oxidizing . . . contains no unsaturated soaps

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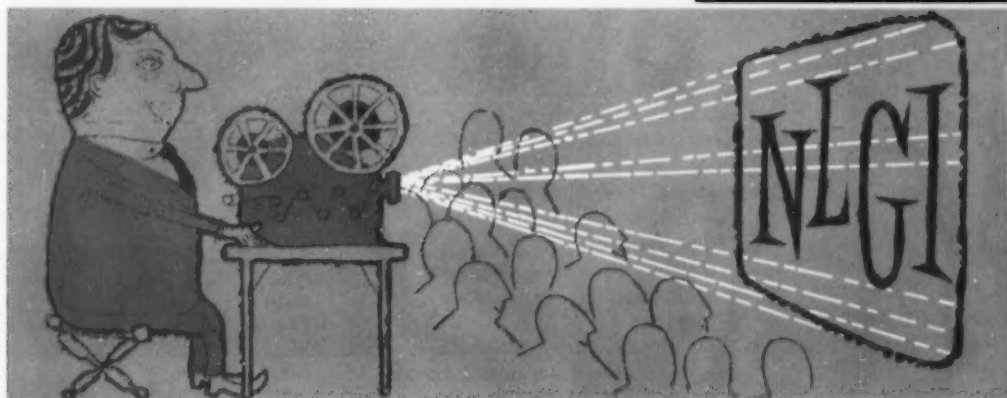
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CHEMICAL COMPANY**

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coming soon ... to all
members—the chance
to buy a selling tool ...



the NLGI-sponsored film on
lubricating greases is a . . .

\$30,000 bargain for \$800!

ORDERS ARE BEING taken now for future delivery on a new 16mm sound film in color—26 minutes of telling and selling the lubricating grease story for members of the NLGI. Costing the Institute over \$30,000 to produce, copies will be sold for \$800 if ordered in advance. Your print can be a valuable public relations feature, training film and educational workhorse for you and your firm. A resume and order card will be mailed to you soon.

21-22 Western Petroleum Refiners Association (regional meeting), Broadview Hotel, Wichita, Kansas.

24-25 Canadian Gas Association, Manoir Richelieu, Murray Bay, Quebec, Canada.

24-27 Michigan Gas Association, Grand Hotel, Mackinac Island, Mich.

25-29 American Institute of Electrical Engrs. (1956 Summer & Pacific general), San Francisco, Calif.

28-30 Rocky Mountain Oil and Gas Association (midyear meeting), Utah Hotel, Salt Lake City.

JULY, 1956

10-12 API Committee on Agriculture (Summer business meeting and annual field trip), College of Agriculture, Cornell University, Ithaca, N. Y.

18-21 Silver Bay Conference on Human Relations in Industry, Silver Bay, N. Y.

AUGUST, 1956

16 API OIIC Steering Committee, API Board Room, New York City

19-24 National Congress of Petroleum Retailers, Inc. (10th annual session), Shoreham Hotel, Washington, D. C.

SEPTEMBER, 1956

6-7 API Oil Industry Information Committee, Conrad Hilton Hotel, Chicago.

7 Midwest Research Institute Symposium on Industrial Development, Linda Hall Library, Kansas City, Mo.

7-8 Desk & Derrick Club, New Orleans, La.

12-14 National Petroleum Association (annual meeting), Traymore Hotel, Atlantic City, N. J.

13-14 Petroleum Packaging Committee, Palmer House, Chicago, Illinois.

16-18 New Mexico Petroleum Industries Committee (annual convention), Hilton Hotel, Albuquerque.

16-21 American Chemical Society (130th annual meeting), Atlantic City, N. J.

16-22 ASTM 2nd Pacific Area National Meeting and Apparatus Exhibit, Hotel Statler, Los Angeles, Calif.

19-21 National Industrial Conference Board (marketing meeting) Waldorf-Astoria Hotel, New York, N. Y.

20-21 Western Petroleum Refiners Association (technical industrial relations meeting), Henning Hotel, Casper, Wyo.

20-21 Mid-Continent Oil & Gas Assn. (membership meeting La.-Ark. Division), Roosevelt Hotel, New Orleans, La.

24-25 IOCA Ninth Annual Meeting, Bismarck Hotel, Chicago, Ill.

OCTOBER, 1956

1-3 Texas Mid-Continent Oil & Gas Association (annual meeting), Rice Hotel, Houston, Texas.

2-3 Texas Mid-Continent Oil and Gas Association (37th annual meeting), Rice Hotel, Houston.

2-4 National Association of Corrosion Engineers (South central region), Beaumont, Texas.

2-6 Society of Automotive Engineers, Inc. (national aeronautic meeting, aircraft enrg. display), Statler Hotel, Los Angeles.

3 American Iron and Steel Institute (regional technical meeting), Thomas Jefferson Hotel, Birmingham.

1-5 American Institute of Electrical Engrs. (1956 Fall general), Morrison Hotel, Chicago, Ill.

7-9 American Association of Oilwell Drilling Contractors (annual meeting), Texas Hotel, Fort Worth.

8-10 The American Society of Mechanical Engineers (joint ASME-ASLE lubrication conference), Chalfonte - Haddon Hall, Atlantic City.

22-24 NLGI ANNUAL MEETING, Edgewater Beach Hotel, Chicago, Ill.

22-24 American Standards Association (7th national conference on standards), Roosevelt Hotel, New York City.

22-24 Rocky Mountain Oil and Gas Association (annual convention), Cosmopolitan Hotel, Denver.

HARSHAW LEAD BASE

Harshaw Lead Base, as an additive to petroleum lubricants, improves extreme pressure characteristics and imparts the following desirable properties:

Increased film strength

Increased lubricity

Improved wetting of metal surfaces
A strong bond between lubricant and metal surfaces

Resistance to welding of metals at high temperatures

Moisture resistance and inhibits corrosion

Harshaw Lead Bases are offered in three concentrations to suit your particular needs:

Liquid	Liquid	Solid
30% Pb	33% Pb	36% Pb

Other metallic soaps made to your specifications. Our Technical Staffs are available to help you adapt these products to your specific needs.

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Everything that moves
DEPENDS ON GREASE!

Almost everything that moves either in actual operation or in the process of its making . . . from gate hinges to tractor wheels . . . depends upon grease. That is why lubricants should be bought with care. You can always depend upon Deep Rock highest quality greases and lubricants. They are manufactured to give top lubrication to all moving parts.

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This will
TAKE THE "SQUEAK"
OUT OF
AMERICAN CARS
for a
Long Time



Shortly after this picture was taken, tons of lithium ore were processed into lithium hydroxide and sent to the grease industry for the production of more lithium base multipurpose grease. And this scene will be repeated over and over and over again. Foote Mineral's vast reserves of lithium ore will meet the ever increasing demands of the grease industry for many, many years to come.

Copies of an informative article on the manufacture and use of lithium base multipurpose grease are available upon request.



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An entirely new development in more efficient, lower cost equipment for finishing greases after manufacture and milling. Brings all the advantages of the Stratco lube oil Flash Evaporator to the grease industry.

Exposes high velocity thin film of grease in vacuum zone for efficient release of water vapor and gases. Adjustable to almost any desired degree of dehydration and to complete deaeration. Nominal capacity 50 G.P.M. and adaptable to either batch or continuous manufacture.

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